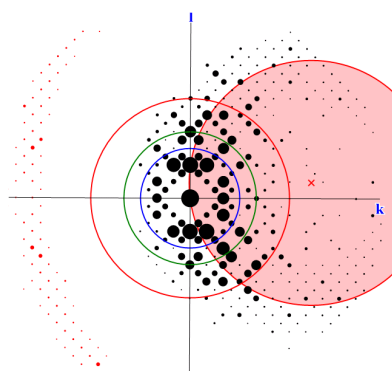


# Simulator



## User's Manual

**Version: 2.0**

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June 2008

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Simulator is part of the eMap & Simulator suite of programs for advanced calculations in electron crystallography.

# 1. Installation

Simulator runs under Windows ® 2000, XP and Vista. About 65MB of hard disk space is needed for the whole package (eMap and Simulator) programs.

**NOTE:** Windows ® 2000 users may need to install Windows Installer 3.1 (available on the CD). The latest redistributable version of Windows Installer is available from the [www.microsoft.com](http://www.microsoft.com) web site or from the installation CD (file: *WindowsInstaller-XXXXXX.exe*). Both eMap and Simulator require MSXML (Microsoft XML engine). The redistributable of MSXML is available from the installation CD (file: *msxml.msi*) or from [www.microsoft.com](http://www.microsoft.com).

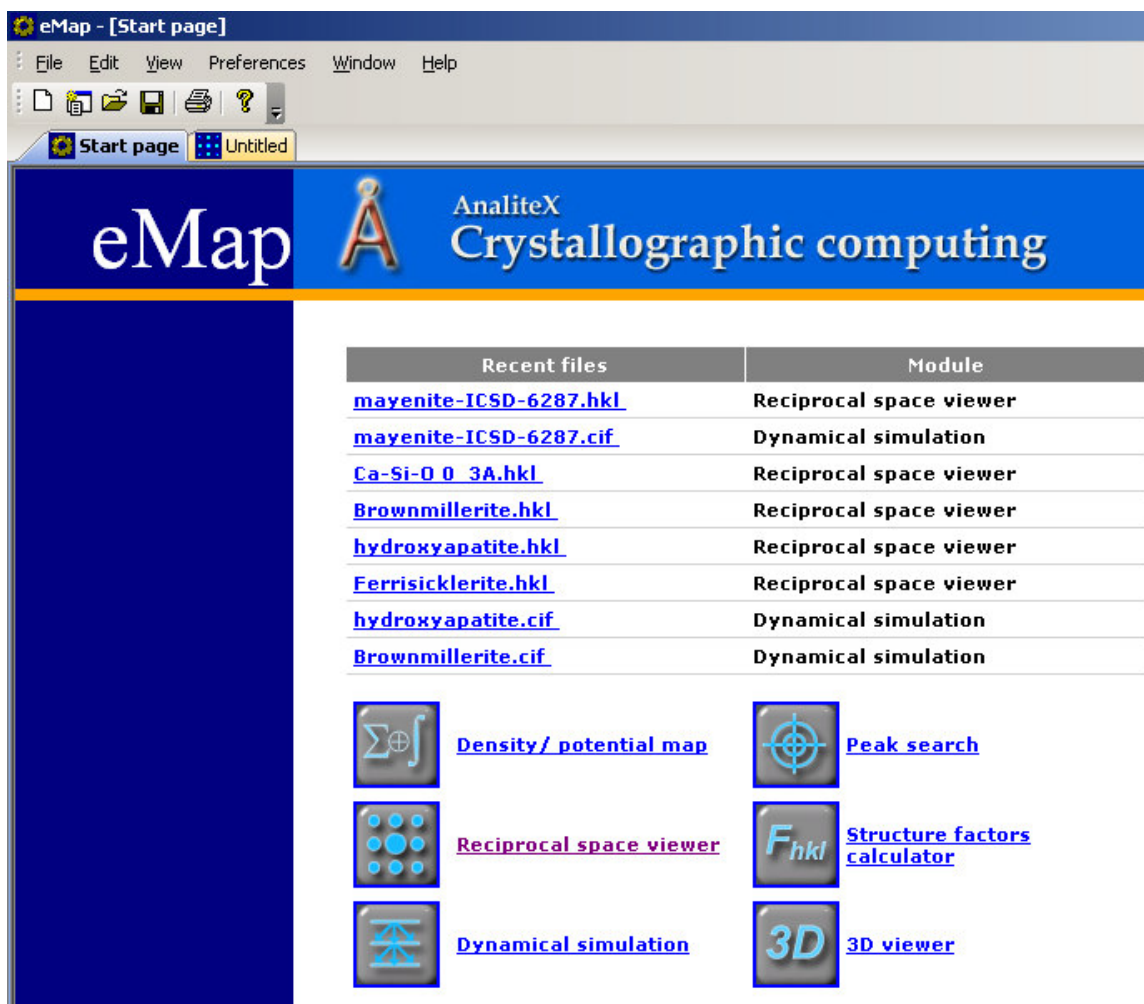
If you have an old version of eMap/Simulator you must deinstall that first, before installing a new version.

Install the program by clicking on **Setup.exe**  located in the directory eMap on the CD. The program will ask you to choose destination location, the default is **C:\Program Files\AnaliTEX\eMap**. Use **Browse** if you want to put the program in another directory, or on another drive. When the directory and drive are as required, click **Next**. Then you will be asked to select program folders under which eMap is run from the Start menu. Select the program folder (default = eMap) and click on **Finish**.

Copy the manual (Multislice simulator 1.0 manual.pdf) from the directory eMap on the CD into the directory to where eMap.exe is located.

## 2. User interface

The Multislice Simulator as any other processing module can be started from the **Start page** by clicking on the **Dynamical simulation** link. **NOTE:** This page will only appear if the *MS Internet Explorer* is installed. In case eMap will fail to locate the Internet Explorer then the simplified **Installed components** dialog will appear.



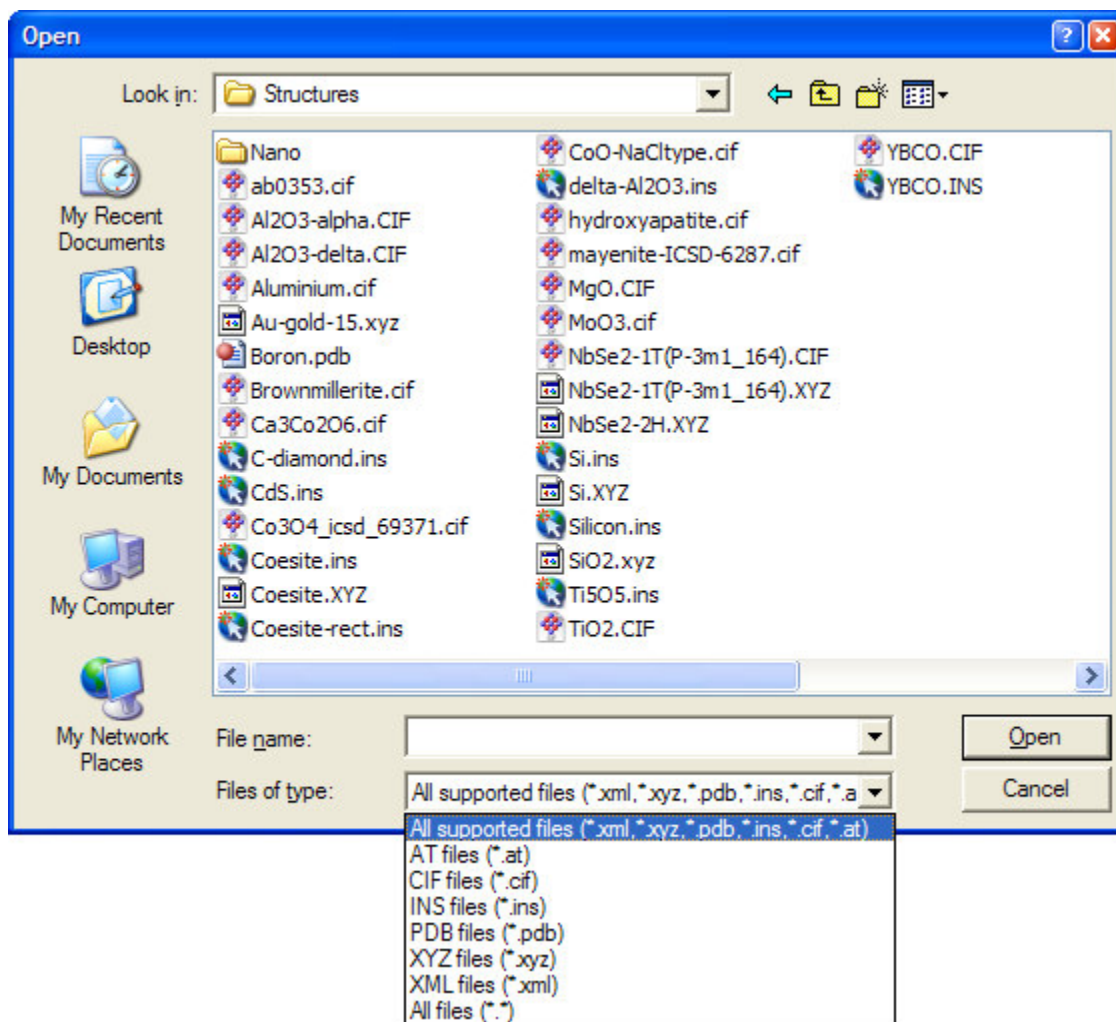
### 2.1. What can we do using Simulator?

Simulator provides you with a great possibility of calculating and displaying different types of information needed in the area of Transmission Electron Microscopy (TEM). The following set demonstrates some of the features that Simulator offers:

- Projected potential;
- Exit wave function;
- Electron Diffraction (ED) patterns;
- High Resolution Electron Microscopy (HREM) images.

## 2.2. Loading the structural information

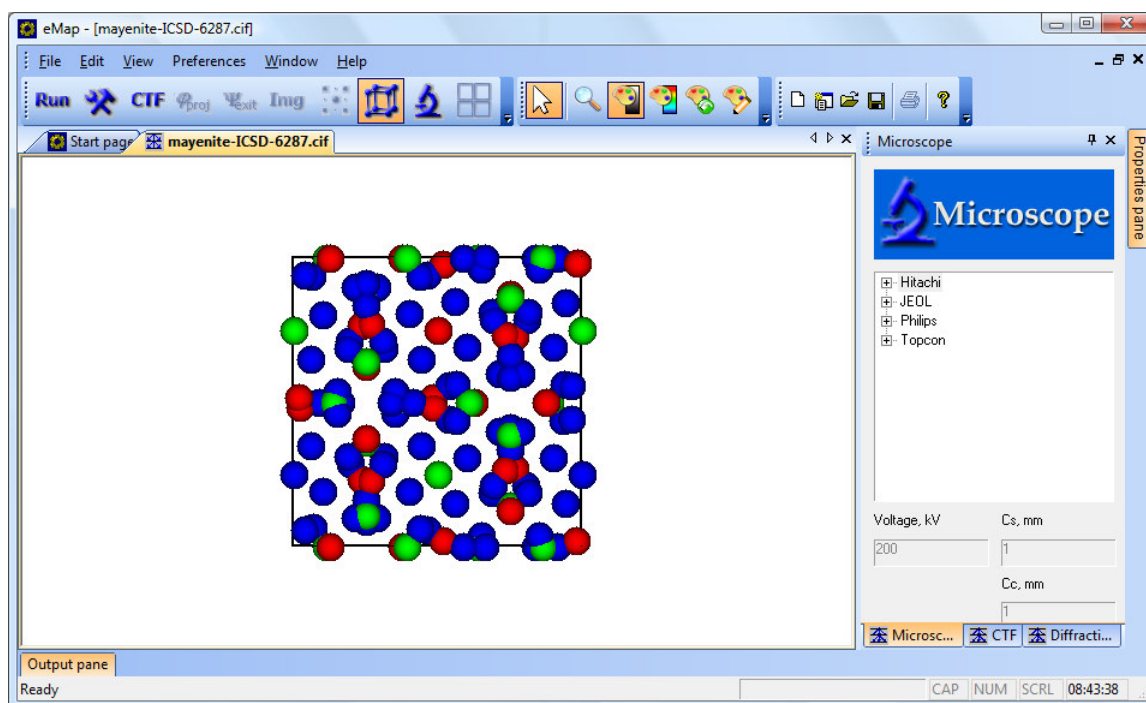
The most commonly used file formats are available to you in the Simulator. You can open PDB, CIF, INS, and XYZ formatted files, plus an XML formatted file used by Simulator to store the structural information as well as calculation settings.



Simulator will start with an empty page if you click on the **Dynamical simulations** link on the **Start page**. In this case you should open your file with structural information. If you can find the file you previously opened on the **Start page** then you can open it directly in Simulator by clicking on the file name in the **Recent files** column of the **Start page**. In this case Simulator will load and display the crystal structure read from your file.

## 2-3 Contrast Transfer Function view

Open the file *Mayenite-ICSD-6287.cif* and you will see this:



### 2.3. Toolbars

The following two toolbars are available when Simulator starts: **Multislice toolbar** and **Image properties toolbar**. If you observing an electron diffraction pattern then the third toolbar becomes visible (Reciprocal space viewer, see **Reciprocal space viewer** Chapter 9 for details).

If no previously calculated data is available, then the Multislice toolbar will have the following appearance



Some of the buttons are disabled.

Some buttons become available only after some calculations have been done. These buttons are also available when you save your calculation data on the disk. In this case Simulator checks these files during start and loads the corresponding data from disk.



Most of the buttons are enabled.

## 2.4. *Multislice toolbar buttons description*



Calculation button (Run). Performs the multislice calculation with given settings (see chapter 6).



Change settings button. Modify most of the multislice settings here (see chapter 5).



Contrast Transfer Function (CTF) view. Modify some settings of CTF and observe the CTF curve in real time for the selected microscope (see chapter 4).



Projected potential view. Observe the calculated projected potential (see section 7.2).



Exit wave function view. Observe the calculated exit wave functions for selected thickness values (see section 7.3).



High resolution electron microscopy (HREM) image. Observe the calculated HREM images for selected thickness and defocus values (see section 7.4).



Electron diffraction pattern. Observe the calculated electron diffraction patterns based on the calculated Exit wave functions for selected thickness values (see section 7.6).



Crystal structure view (*for current zone axis only*). See the structure from the present direction (zone axis).



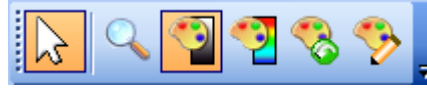
Microscopes docking pane. Specify the electron microscope to be used in the simulation (see section 3.1).



Adjacent cells dialog. Select the required number of adjacent unit cells (see section 7.1).

## 2.5. Working with the image toolbar

The second available toolbar in Simulator allows working with calculated images. See Chapter 7 for full details.



Standard arrow pointer.



### **Magnifying glass – zooming in and out**

*Zooming in* is done using the **Magnifying glass** on the toolbar and then clicking anywhere within the image.

**NOTE:** the cursor will change from standard to the magnifying glass with a '+' symbol inside.

*Zooming out* can be done using the same toolbar button and holding the **CTRL** keyboard button while pressing the left mouse button within the view.

**NOTE:** the cursor will change from standard to the magnifying glass with a '-' symbol inside.



Grayscale palette. Sets the colors of the current image view into grayscale.



Color palette. Sets the color palette of the current image view.



Resets the palette sliders into their initial positions.

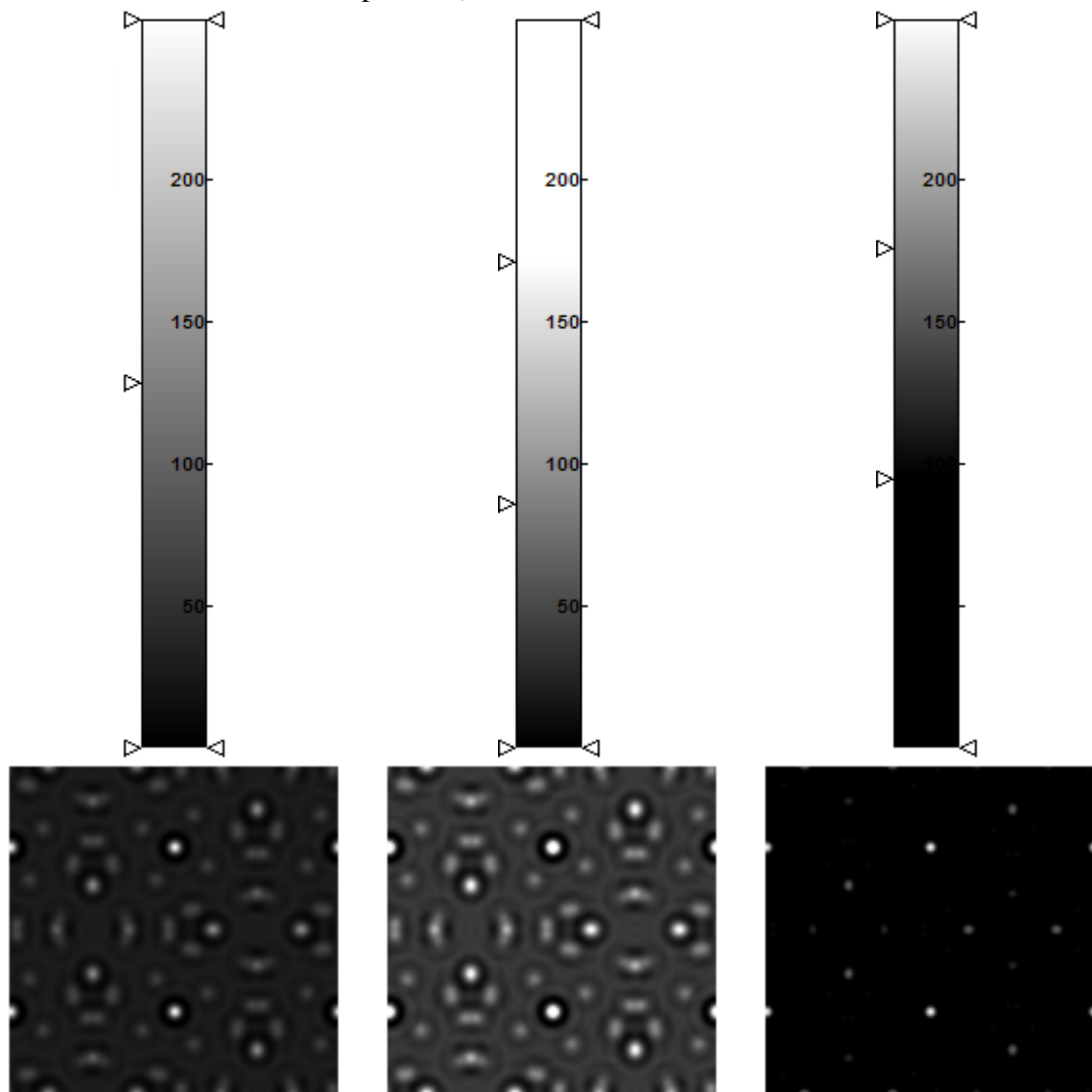


Provides the possibility to modify the color palette. **NOTE:** not available in the current release of the Simulator.

## 2.6. *Palette control*

There is a special palette control available in any of the image view modes. This control helps in modifying the brightness and contrast values of all images in the current view. Visible in Projected potential (Chapter 7.2), Exit wave function (Chapter 7.3) and HREM image views (Chapter 7.4).

The following 3 screenshots show the way of changing the brightness and contrast by dragging one of 3 sliders on the left side of the control. The slider in the middle remains equidistant from the upper and lower sliders when dragging one of these two sliders. During dragging the middle slider, the program tries to keep distances to upper and lower sliders equal (except in the cases when upper slider reaches the upper bound and then remains at the same position or when the lower slider reaches the bottom bound and then remains at the same position).

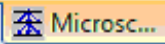
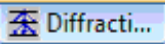


Neutral positions of palette sliders.

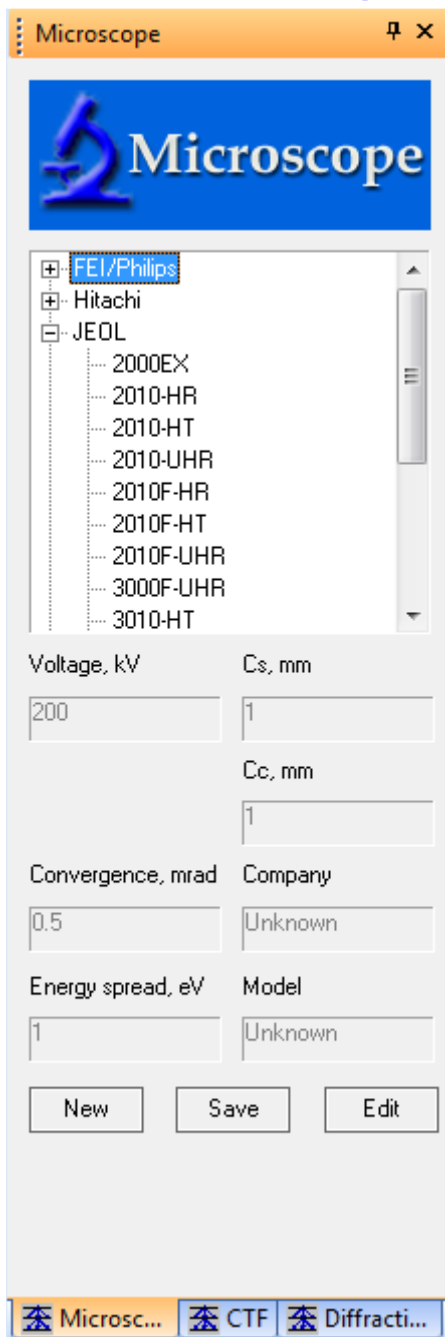
Dragging the upper slider down.

Dragging the bottom slider up.

### 3. Docking panes

This chapter describes the available *Docking panes*   . They can be used when simulating HRTEM images and electron diffraction patterns, as described in Chapter 4.

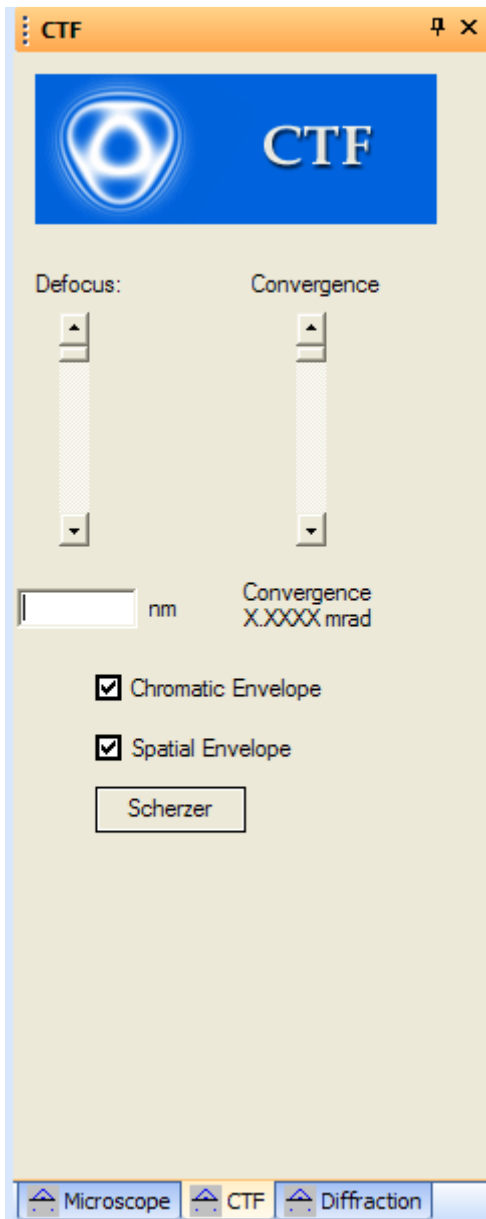
#### 3.1. Microscope docking pane



This pane is designed to work with microscopes. The user can:

- Browse microscopes in the Tree control;
- Edit selected microscope parameters;
- Create a new microscope with specified parameters;
- Save the existing updates.

### 3.2. Contrast Transfer Function (CTF) docking pane

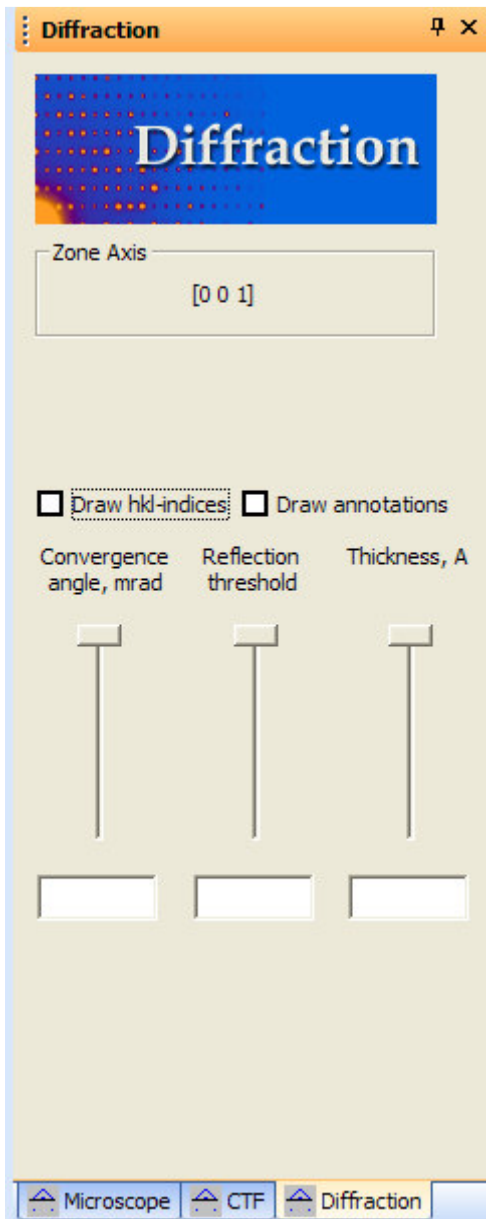


This pane is designed to work with the parameters of the Contrast Transfer Function (CTF). The user can:

- Change the defocus value by dragging the **Defocus** slider;
- Manually edit the defocus value by typing in the edit box under the **Defocus** slider. In order to apply changes, the user must do a mouse-click outside the edit box. The defocus values are in [nm];
- Change the convergence angle value by dragging the **Convergence** slider;
- Show/hide the **Chromatic** and **Spatial Envelopes** by marking/unmarking the corresponding check boxes;
- Set the defocus value to the Scherzer defocus by pressing the **Scherzer** button.

All changes will be applied to the CTF plot in run-time (see section 4). However, they will not affect the image simulations.

### 3.3. Diffraction docking pane

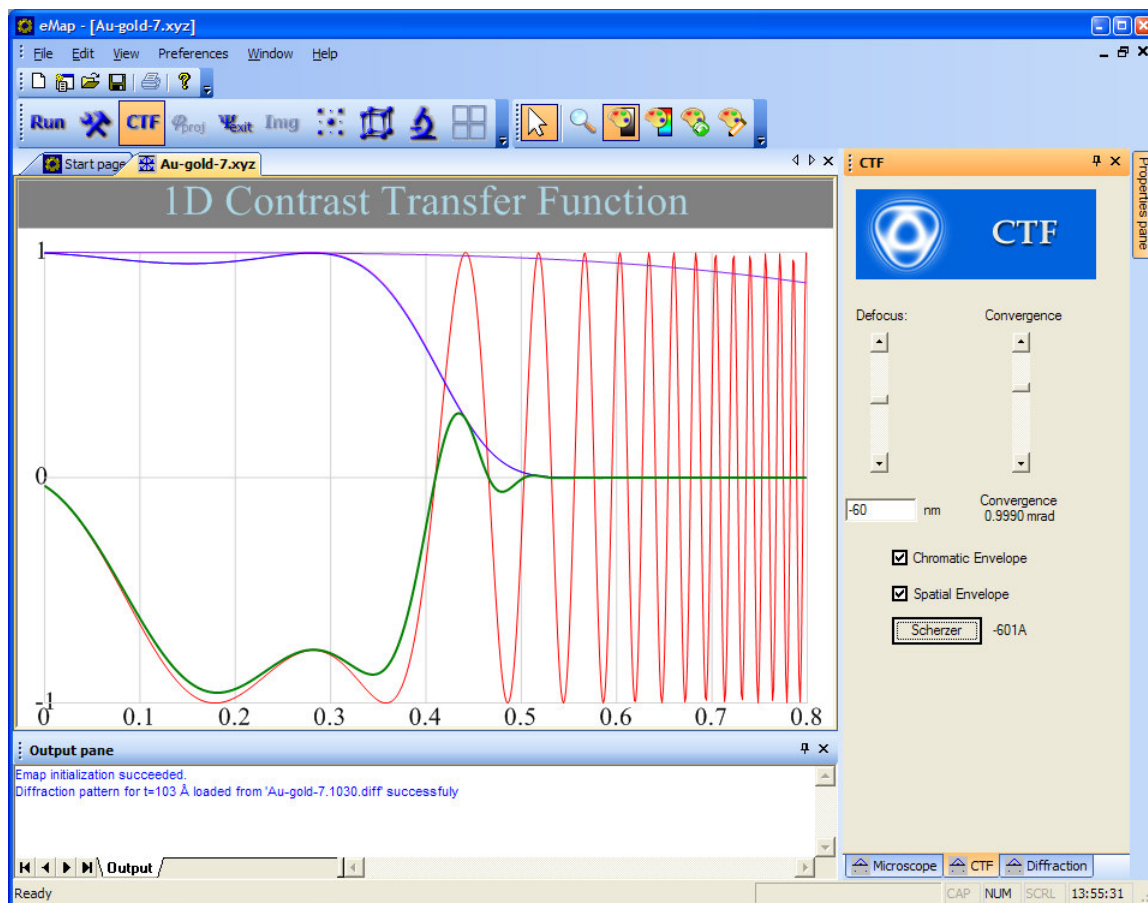


This pane is designed to work with the parameters of the simulated electron diffraction pattern. The user can:

- Draw/hide the *hkl*-indices and text annotations on the diffraction pattern view by marking/unmarking the corresponding check boxes;
- Change the convergence angle (the spot size, in *mrاد*);
- Change the threshold values of reflections to be shown on the diffraction pattern view;
- Change the thickness of the diffraction pattern to be shown. Only diffraction patterns at specified thickness are available (see **Thickness** in the **Zone Axis** property page, section 5-11);
- Manually edit the convergence angle, reflection threshold and thickness values by typing into the corresponding edit boxes under the sliders. In order to apply changes, the user must do a mouse-click outside the edit box. In case the manually modified **Thickness** value doesn't correspond to any specified thickness values, the diffraction pattern at *closest calculated thickness* will be shown.

## 4. Contrast Transfer Function view

The user can observe the Contrast Transfer Function (CTF) and modify the main parameters of the CTF in real time for the selected microscope, using **CTF**. See section 3-2 for the details on the properties of the CTF docking pane.



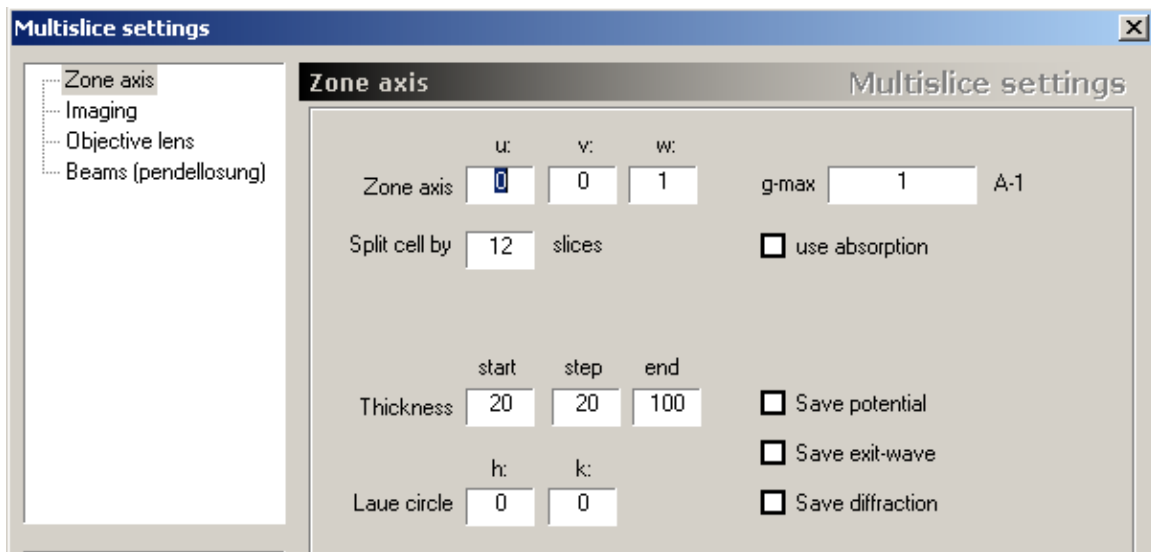
## 5. Multislice settings

This chapter explains the basic settings, which the user can change in order to achieve the required results, using the multislice method implemented in the Multislice Simulator. The dialog with all available settings can be opened using the **Settings**  button on the **Multislice toolbar** (see section 2-4). The **Zone axis** should be activated (default when entering the **Multislice settings**).

### 5.1. Zone Axis settings

This property page can be used for modifying the basic settings of the multislice calculator related to the crystal direction. Among them are:

- The **Zone Axis** – can be changed using 3 indices  $u$ ,  $v$  and  $w$ ;
- The reciprocal resolution **g-max** – limits the total number of beams to be used in the calculations (in reciprocal Ångströms,  $\text{\AA}^{-1}$ ); g-max 2 corresponds to a maximal resolution of 0.5 Å.



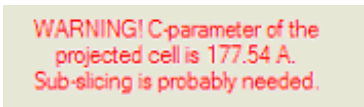
- The number of slices to split the unit cell along the projection direction. Simulator calculates the translation vector along the projection direction and suggests the number of slices per unit cell so that the slice thickness is  $\sim 1 \text{ \AA}$ . The user can modify this number. However, some factors should be considered when choosing other values (see section 6-16 for more details);
- The **Thickness** range (**start** and **end** values) and the **step** between the bounding values;
- Beam tilt off from the specified zone axis can be set through the **Laue circle** position by changing the  $h$  and  $k$  values (can be real numbers) along corresponding 2D axes in reciprocal space. The configuration without any beam tilt corresponds to (00) for  $h$  and  $k$ .

The calculations results will be saved for future use only if the specified check boxes (for example **Save potential**) will be marked. The files with calculation results will be deleted after closing Simulator.

Only images and diffraction patterns for specified thickness values can be shown after the calculations are done. In case with **Thickness start** = 20 Å, **step** = 20 Å and **end** = 100 Å only 5 exit wave functions, 5 electron diffraction patterns and at least 5 rows of HREM images (number of those within each row depends on the number of the specified defocus values) will be stored for reviewing. The corresponding 5 thickness values are 20 Å, 40 Å, 60 Å, 80 Å and 100 Å. The diffraction patterns can only be shown one by one (see Chapter 7.6), while the others can all be seen in Montage mode (see Chapter 7.5), i.e. all at the same time.

**NOTE:** The use of absorption during the structure factors calculation *is not* available in the current version of Simulator.

The slicing scheme used in the current version of Simulator assumes that if the cell extension in the projection direction is larger than 20Å, the user should consider using the 3D potential. In this case a **WARNING** will appear as this:

A rectangular box with a light beige background and a thin black border. It contains three lines of red text.

WARNING! C-parameter of the  
projected cell is 177.54 Å.  
Sub-slicing is probably needed.

**NOTE:** the 3D potential calculations are not available in the current version of Simulator.

## 5.2. Imaging

The Imaging property page allows the user to control the settings for the HREM images during the multislice calculations. Click on settings  and then .

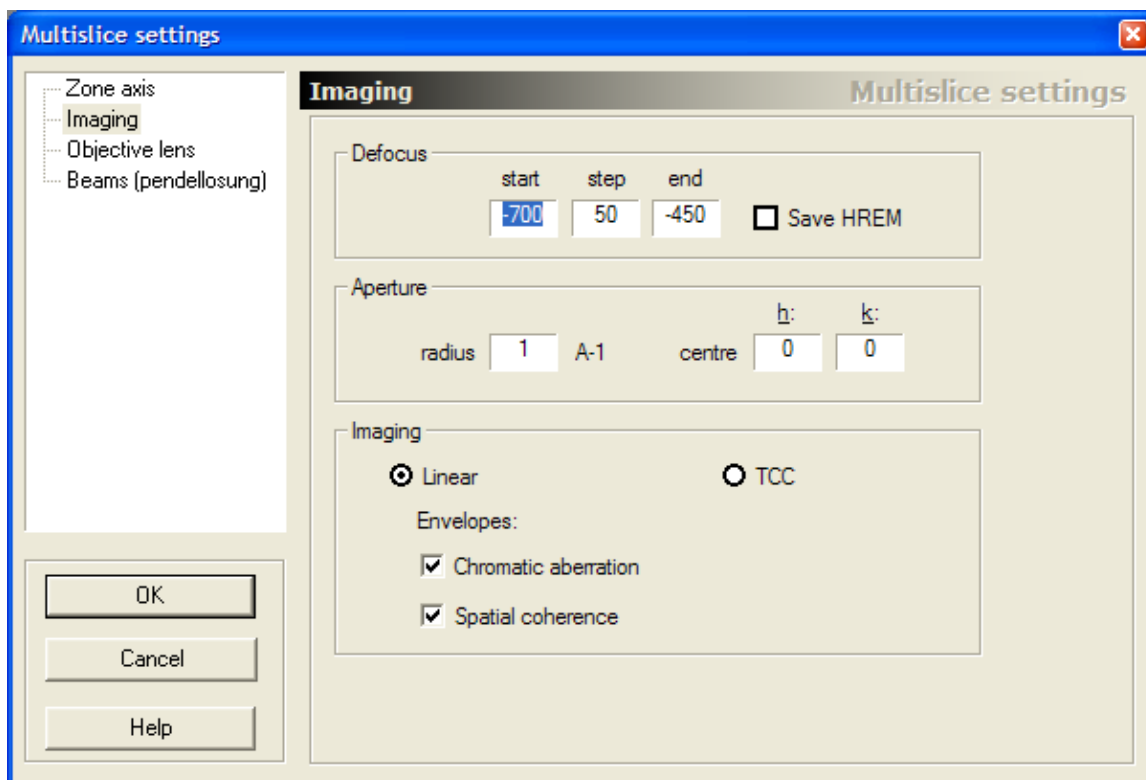
### 5.2.1. Defocus

The defocus range can be assigned by selecting the starting point (**start**), the finish (**end**) and the step between **start** and **end**. If only one defocus value should be calculated then the **start** value must be equal to the **end** value. If the **step** is set to 0 and the **start** is different from the **end**, then only 2 defocus values will be calculated.

The **Save HREM** check box sets the flag that indicates if the calculated HREM images should be saved in the corresponding files for later use.

### 5.2.2. Aperture

The **aperture** radius value is in reciprocal Ångström ( $\text{\AA}^{-1}$ ). It is possible to specify the centre of the aperture by changing the **centre h** and **k** values. **NOTE:** These values can be real. The (00) setting corresponds to the aligned aperture position.



### 5.2.3. Linear imaging

Finite energy spread envelope function  $E_c$  (chromatic aberration envelope):

$$E_c(u) = \exp\left(-\frac{1}{2}(\pi\lambda\delta)^2 u^4\right)$$

where  $\delta$  is the defocus spread.

Finite size of the electron source  $E_s$  (spatial coherence envelope):

$$E_s(u, \Delta f) = \exp\left\{-\left(\pi s_0 \lambda\right)^2 u^2 \left[\Delta f + C_s \lambda^2 u^2\right]^2\right\}$$

where  $\Delta f$  is the defocus value,  $s_0$  is the convergence angle,  $C_s$  is the spherical aberration.

Perfectly coherent illumination transfer function

$$T(u, \Delta f) = A(u) \exp[-i\chi(u, \Delta f)] E_s(u, \Delta f) E_c(u)$$

Scherzer defocus

$$\Delta f_{\text{Scherzer}} = -1.2\sqrt{C_s \lambda}$$

Lichte defocus

$$\Delta f_{\text{Lichte}} = -0.75 C_s (u_{\max} \lambda)^2$$

where  $u_{\max}$  is the maximum transmitted spatial frequency.

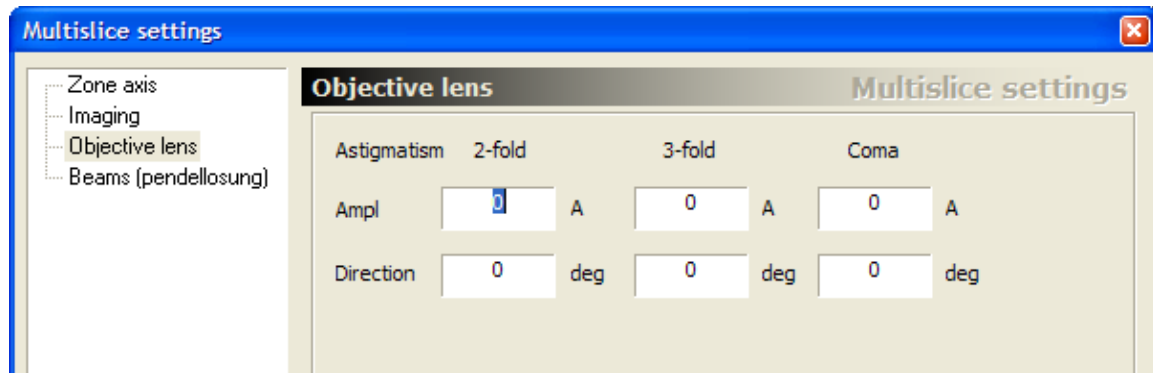
### 5.2.4. Non-linear imaging for partially coherent and incoherent illuminations

Takes place in case when we need to take into account the effects associated with the finite size of the electron source (beam divergence effects) and fluctuations of defocus spread (due to the energy spread of the electron source or instabilities of the objective lens current). In this case the spatial extent of the electron source is large and the image can be considered as incoherent when formed with different defocus and with electrons emitted from different positions on the electron source. The resulting image is given by the average of the set of images formed for all angles of incidence and defocus values.

**NOTE:** The non-linear imaging will be used when TCC is selected in the **Imaging** property page.

### 5.3. Objective lens setup

**NOTE:** Both 2-fold and 3-fold astigmatisms and coma are available only for the Linear imaging in the current version of Simulator. After  click on **Objective lens**.



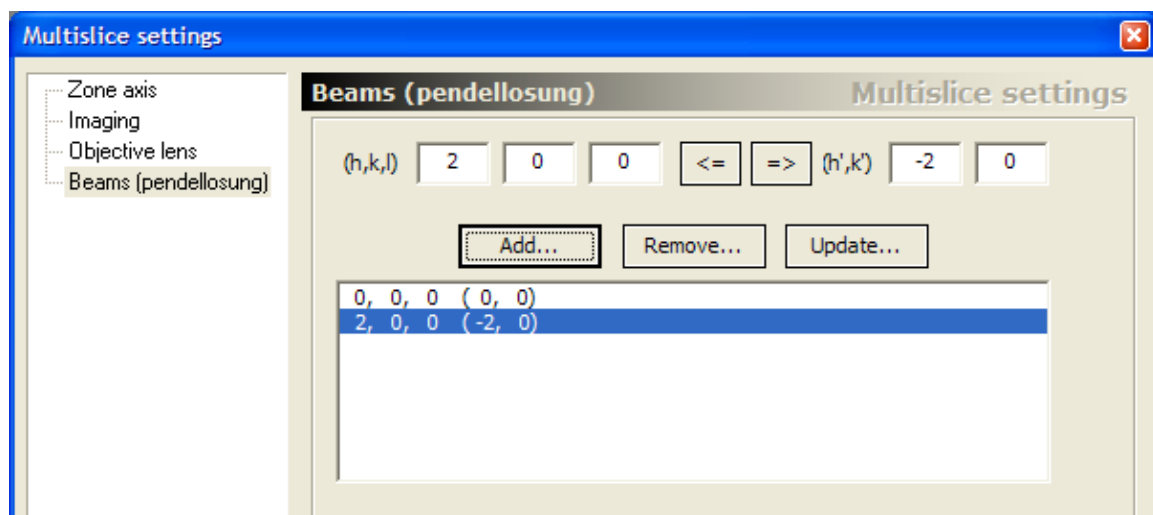
### 5.4. Pendellösung plots

The user can specify the beam(s) the amplitudes and phases of which should be kept during the multislice calculations for every calculated slice. In this case the information will be stored in the file **Pendellosung-XXXX.dat** in the same folder as the loaded structure file. After  click on **Beams (pendellosung)**.

The reflections can be defined in two different ways:

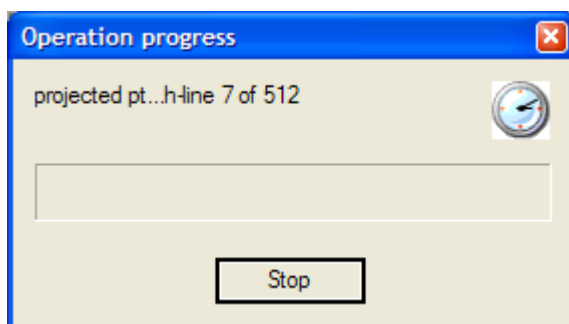
- Defining 3D Miller indices of the reflections by filling in the left 3 boxes with  $h$ ,  $k$  and  $l$  indices. In this case the 3D indices should fit the zone axis equation  $hu + kv + lw = 0$ .
- Defining 2D plane indices in the local axes ( $h'$ ,  $k'$ ).

The transition between indices is possible using  $\leq$  and  $\Rightarrow$  buttons. Indices can be **Added**, **Removed** and **Updated** using corresponding buttons. The data can be viewed using Excell. The format is thickness, Amplitude, Phase (and further pairs of Amplitude, Phase, if more than one beam has been selected). Can be viewed using Excel.

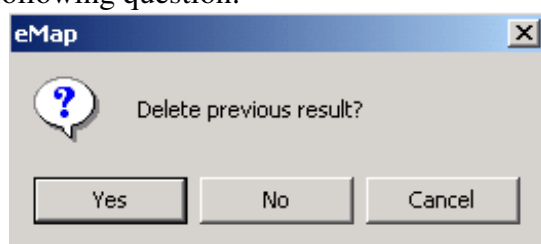


## 6. Multislice calculations

The multislice calculations will start after pressing the **Run** button  (see section 2-4). The following progress dialog will be shown:



If you are working with the same structure file and are trying to repeat the same calculations or calculations with modified settings, then Simulator will ask if you would like to clean up, e.g. by removing files generated by previous **Run**. In this case Simulator will come up with the following question:



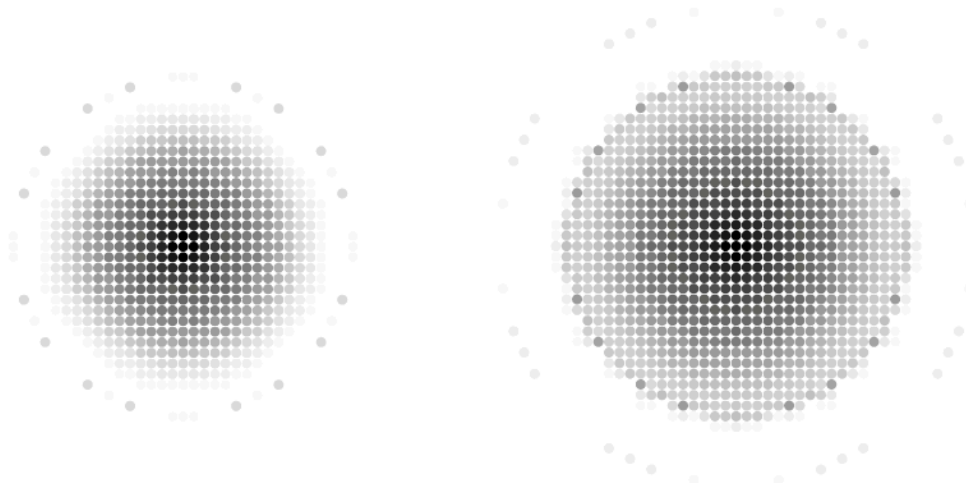
In case of pressing the Cancel button the calculations will be terminated.

Simulator will try to use the data left from any available previous calculations.

### 6.1. Cautions

The major error source in the multislice calculations is the slice thickness ([1] and [2]). The upper limit of the slice thickness can be estimated as  $\Delta z < 2\Delta r^2 / \lambda$  where  $\Delta r$  is the distance within which the potential doesn't change appreciably. In case of 300 kV electrons and  $\Delta r = 0.1 \text{ \AA}$  we have  $\Delta z < 1 \text{ \AA}$ . An acceptable accuracy is typically achieved when the slice thickness is chosen as the radius of an atom, i.e. 1-2  $\text{\AA}$ .

Artificial HOLZ reflections may appear in case the slice thickness is too big or if the resolution is too high.



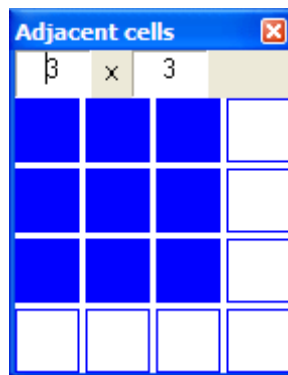
MgO [001] simulated electron diffraction pattern. The contrast of the diffraction pattern to the right is enhanced with respect to the left pattern. The HOLZ rings can be clearly observed. The slice thickness was too big and the selected resolution was too high. This led to the leak of some intensity from ZOLZ into HOLZ which is *incorrect* in this case.

## 7. Reviewing the calculation results

This chapter explains how to inspect the results of calculations using different viewing options. They are activated using any of the options .

### 7.1. *Adjacent cells*

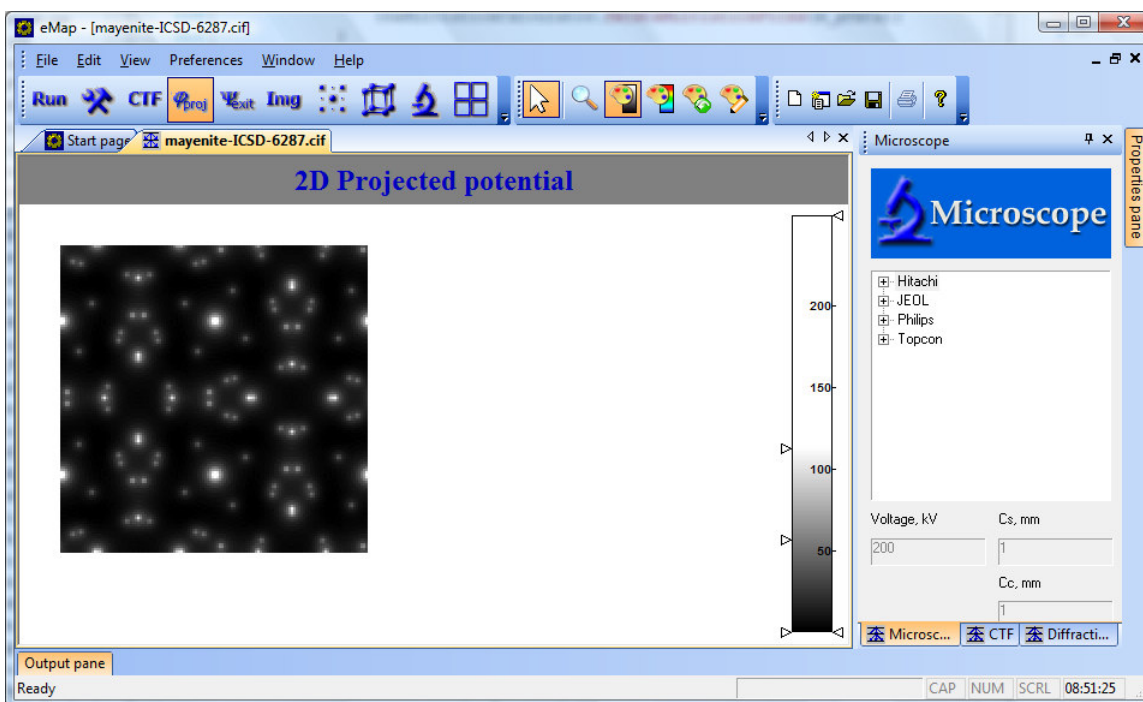
The user can create a view with several cells adjacent one to the other. Default value is 1x1 (single cell). In order to modify the number of adjacent cells, the user should click on the **Adjacent cells** button on the **Multislice toolbar** (see section 2-4). The following dialog will appear. Using the mouse, one can choose the required number of cells in both  $x$ - and  $y$ -directions by holding the left mouse button and moving it across the dialog box. In this case the number of adjacent cells changes dynamically and will be accepted when the user releases the left mouse button. The other way of choosing the number of adjacent cells is by clicking within the corresponding square in the **Adjacent cells** dialog box.



The **Adjacent cells** dialog with 3x3 cells selected.

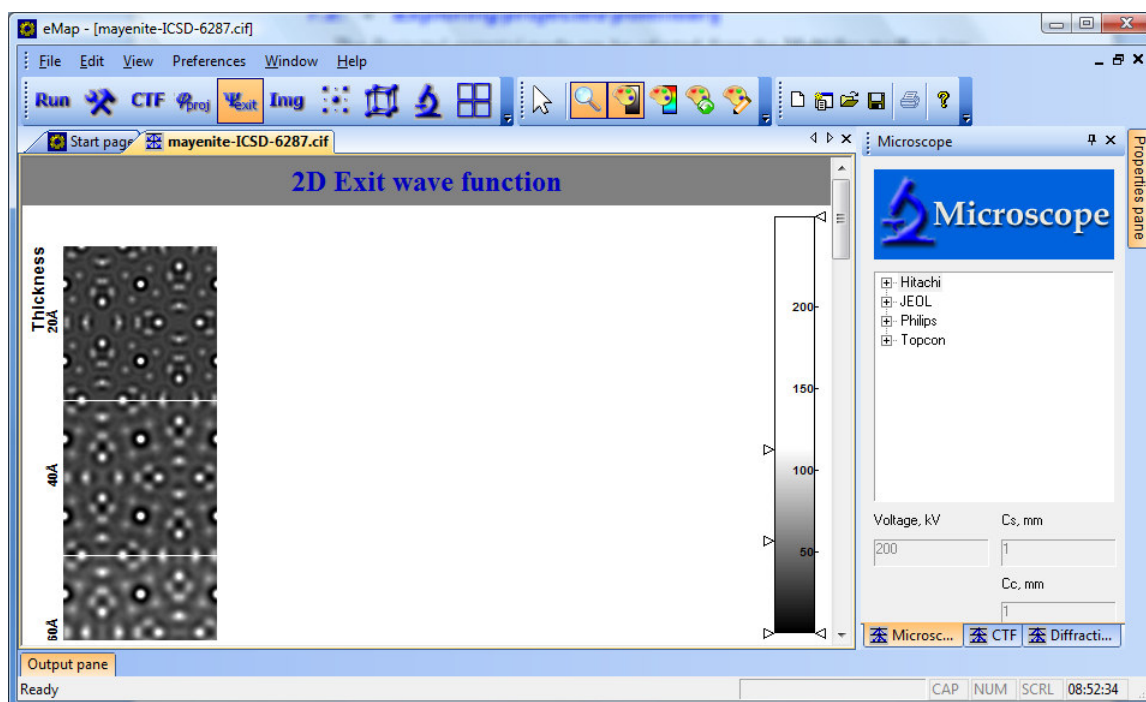
## 7.2. Exploring projected potential

The *Projected potential* mode can be selected, by  from the **Multislice toolbar** (see section 2-4). A single image will always be shown.



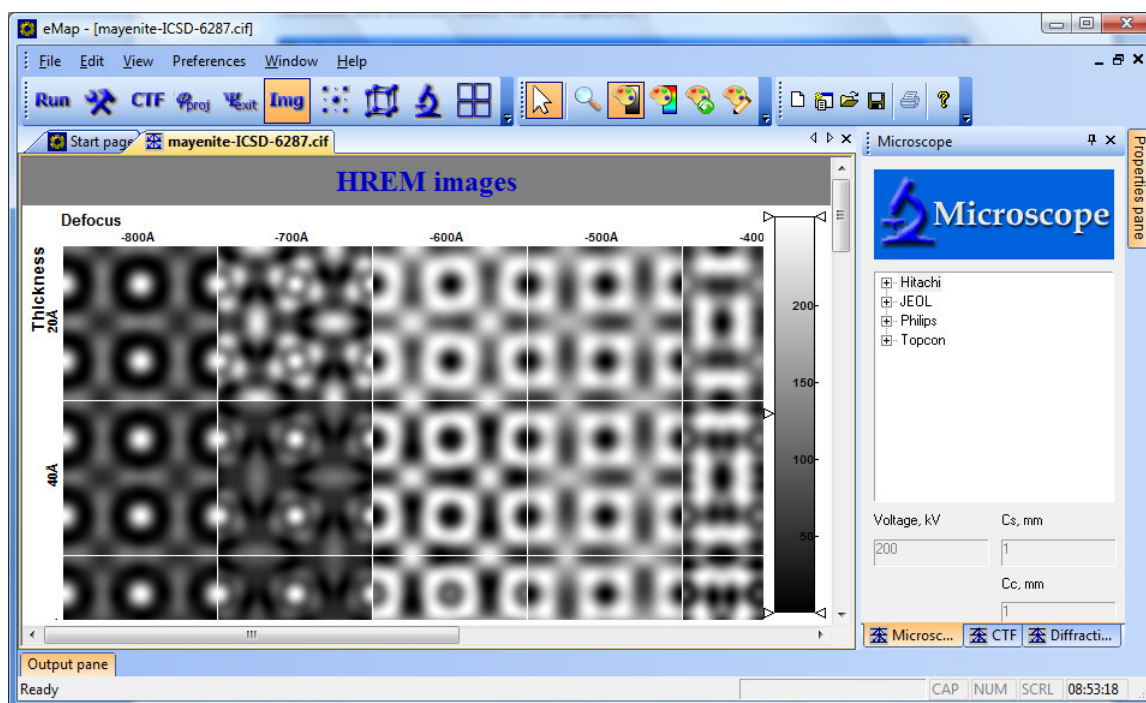
### 7.3. Exploring the exit wave function

The *Exit wave function* mode can be selected by  from the **Multislice toolbar** (see section 2-4). The thickness dependence of the exit wave function is represented vertically starting from the smallest calculated thickness (top) and finishing with the highest thickness (bottom). The user can lower the magnification or use the scroll bars on the sides of the window in order to see all the simulated HREM images. The exit wave functions will be displayed only for specified thickness values. A set of images will be shown, if you have specified a range of focus values, so-called **Montage mode** (see Chapter 7.5).



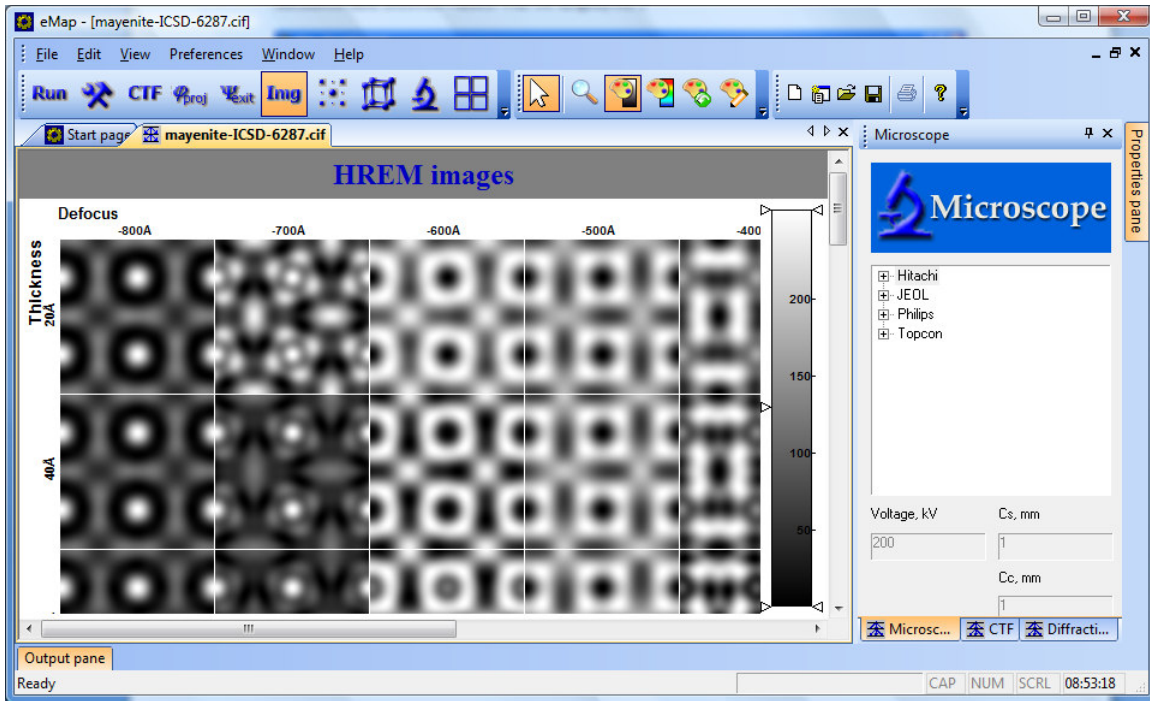
## 7.4. Exploring HREM images

The *HREM images* mode can be selected by **Img** from the **Multislice toolbar** (see section 2-4). The *thickness* dependence of the HREM images is represented vertically starting from the smallest calculated thickness (top) and finishing with the highest thickness (bottom). The *defocus* dependence of the HREM images is represented horizontally starting from the smallest calculated defocus (left) and finishing with the highest defocus value (right). The user can demagnify (by holding down **CTRL** while left-clicking the mouse) or use the scroll bars on the sides of the window in order to see all the simulated HREM images. The HREM images for the specified thickness and defocus values will be displayed, in montage mode (see 7.5).



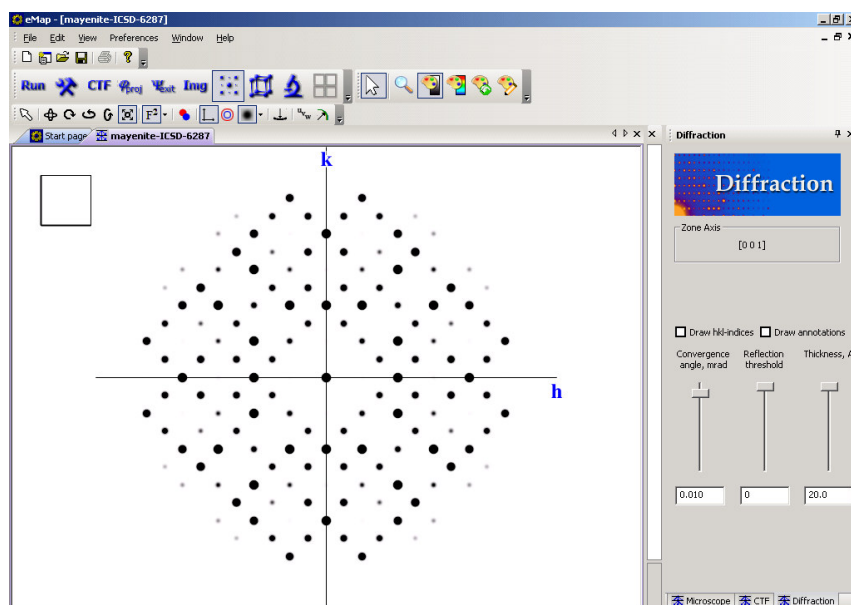
### 7.5. Montage mode

Simulator provides the montage mode to help the user to observe more than one simulated image at the same time. You can use the **Magnifying glass** to *Zoom in* and *out* in order to see more simulated images (see section 2-5 for details on **Magnifying glass**).

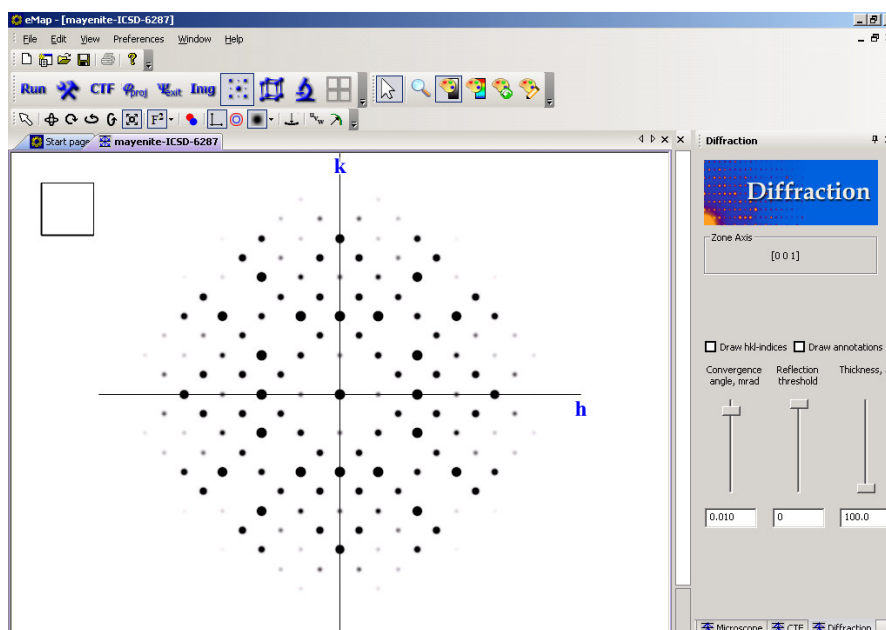


## 7.6. Exploring diffraction

The *Electron diffraction patterns* mode can be selected from the **Multislice toolbar** (see section 2-4). Use the thickness slider on the **Diffraction** pane in order to select the calculated electron diffraction pattern at the required thickness. Shown one by one.



Mayenite electron diffraction patterns. Thickness 20 Å (above) and 100 Å (below).



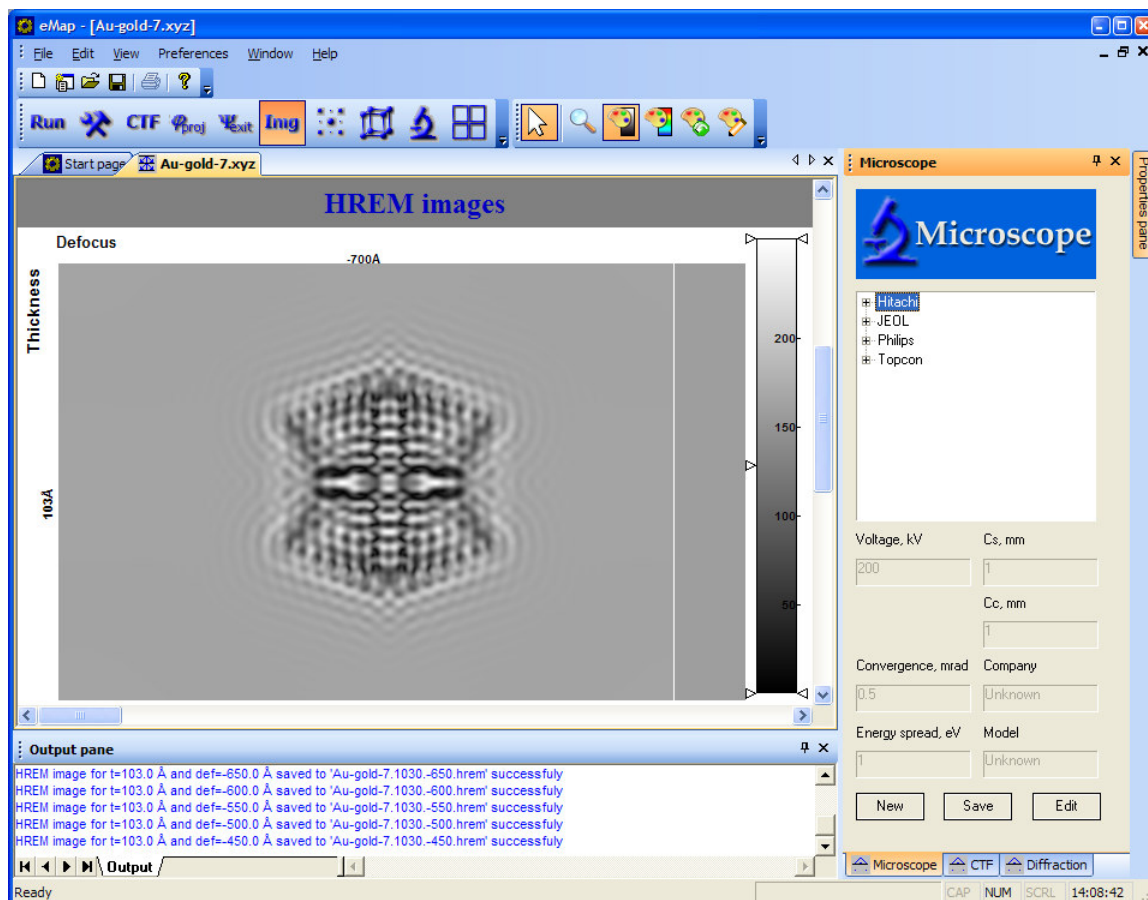
If you specified 5 thickness values in 

|           | start | step | end |
|-----------|-------|------|-----|
| Thickness | 20    | 20   | 100 |

 (see Chapter 5.1), then there will be 5 thickness values available here.

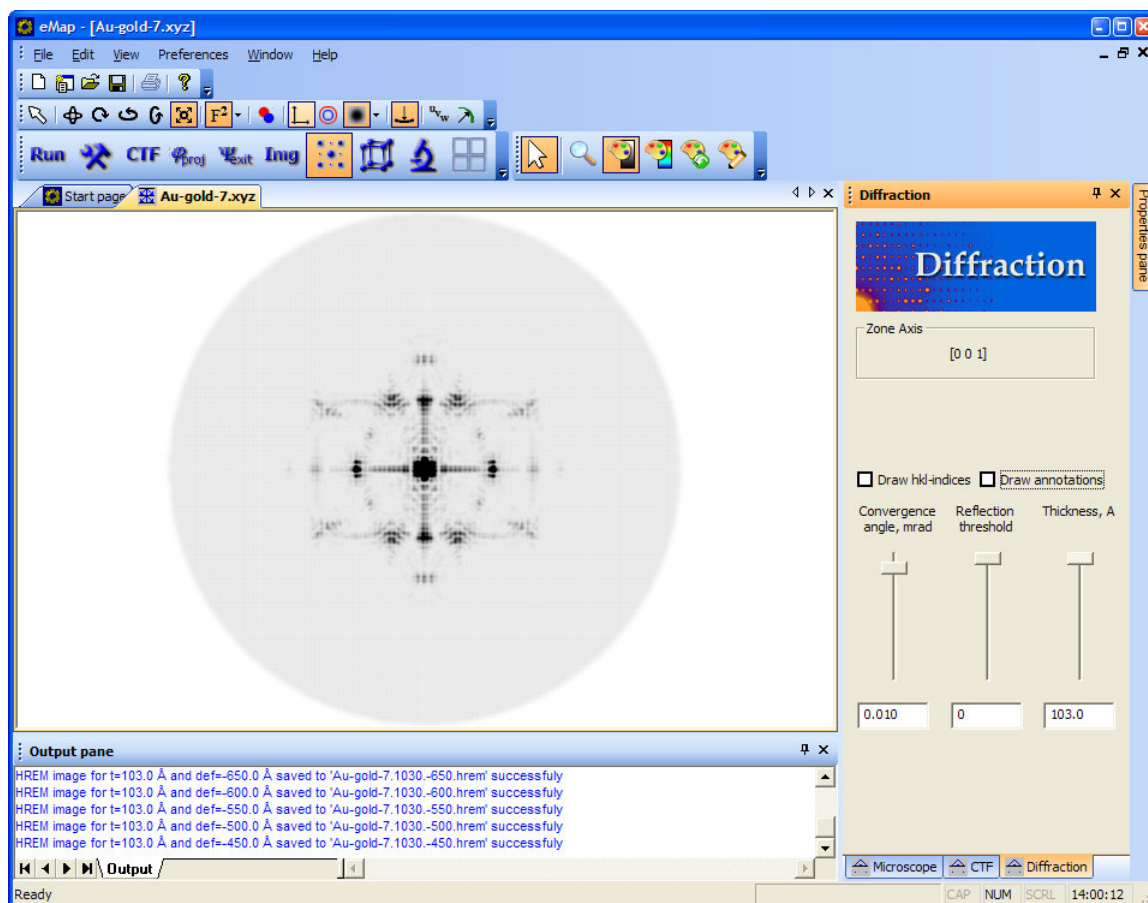
## 8. Working with non-crystalline objects

Simulator is capable to simulate HREM images and electron diffraction patterns for non-crystalline objects and nano-materials. This chapter presents an example of simulations of 7-shell gold nanoparticle.



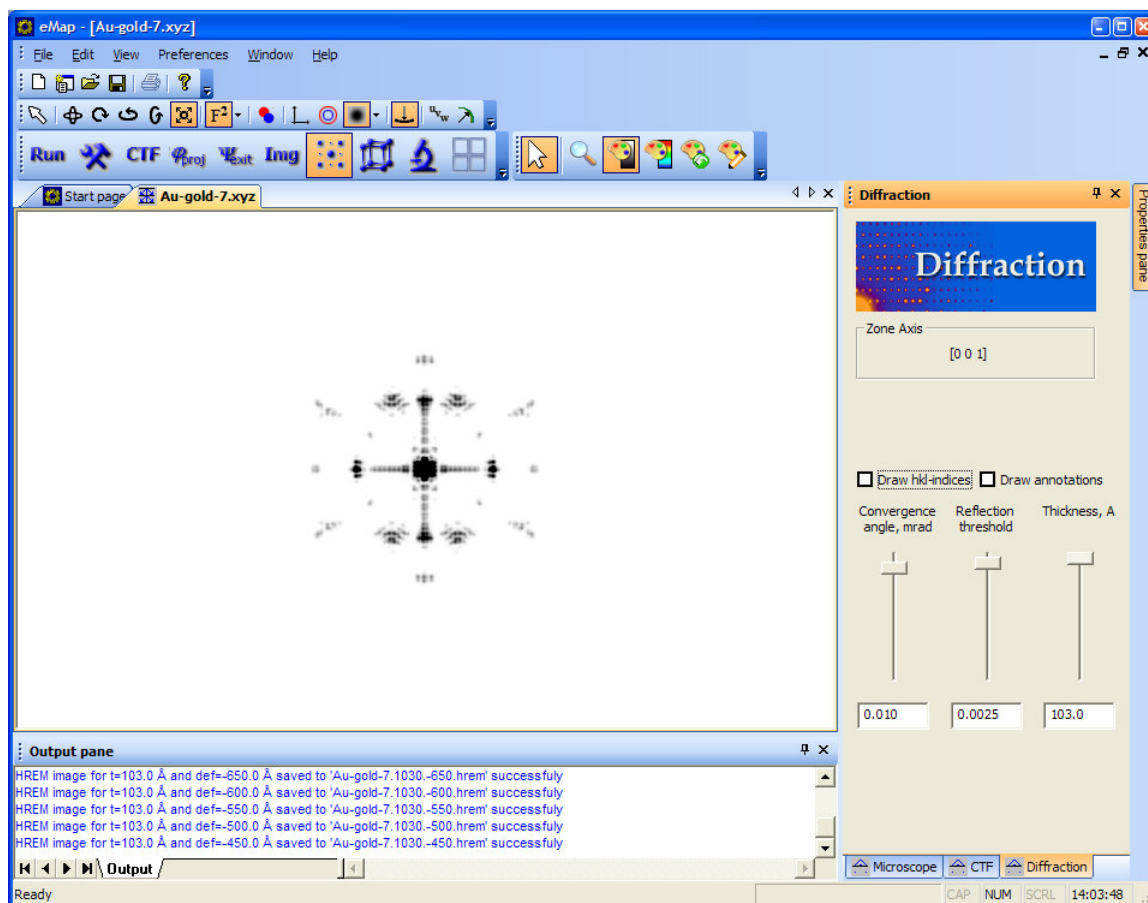
HREM image for defocus  $-700 \text{ \AA}$ .

## 8-2 Working with non-crystalline objects



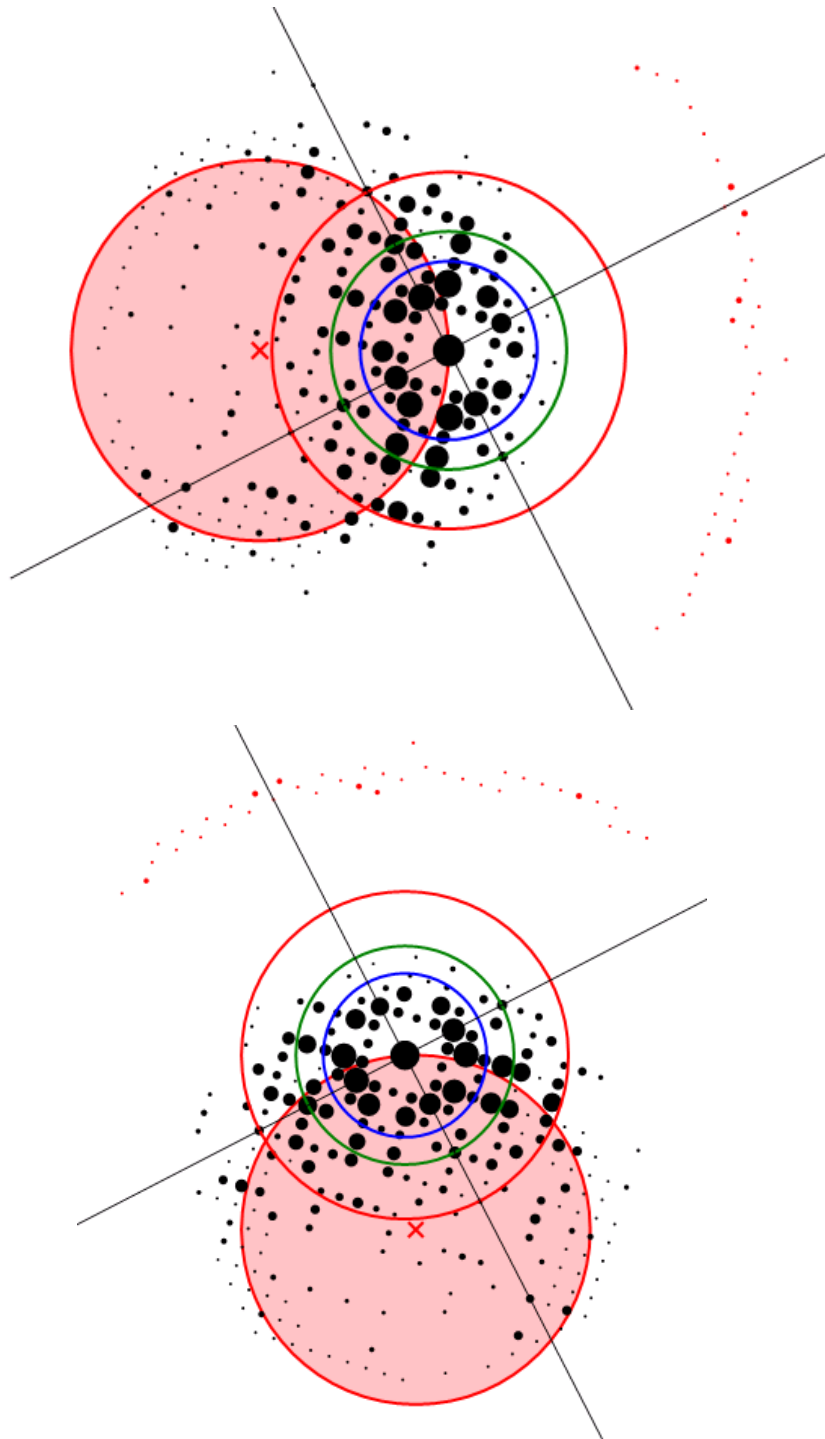
7-shells Au nanoparticle electron diffraction pattern.  
Default settings in the **Diffraction docking** pane.

### 8-3 Working with non-crystalline objects



7-shells Au nanoparticle electron diffraction pattern.  
The Reflections threshold was manually changed to 0.0025.

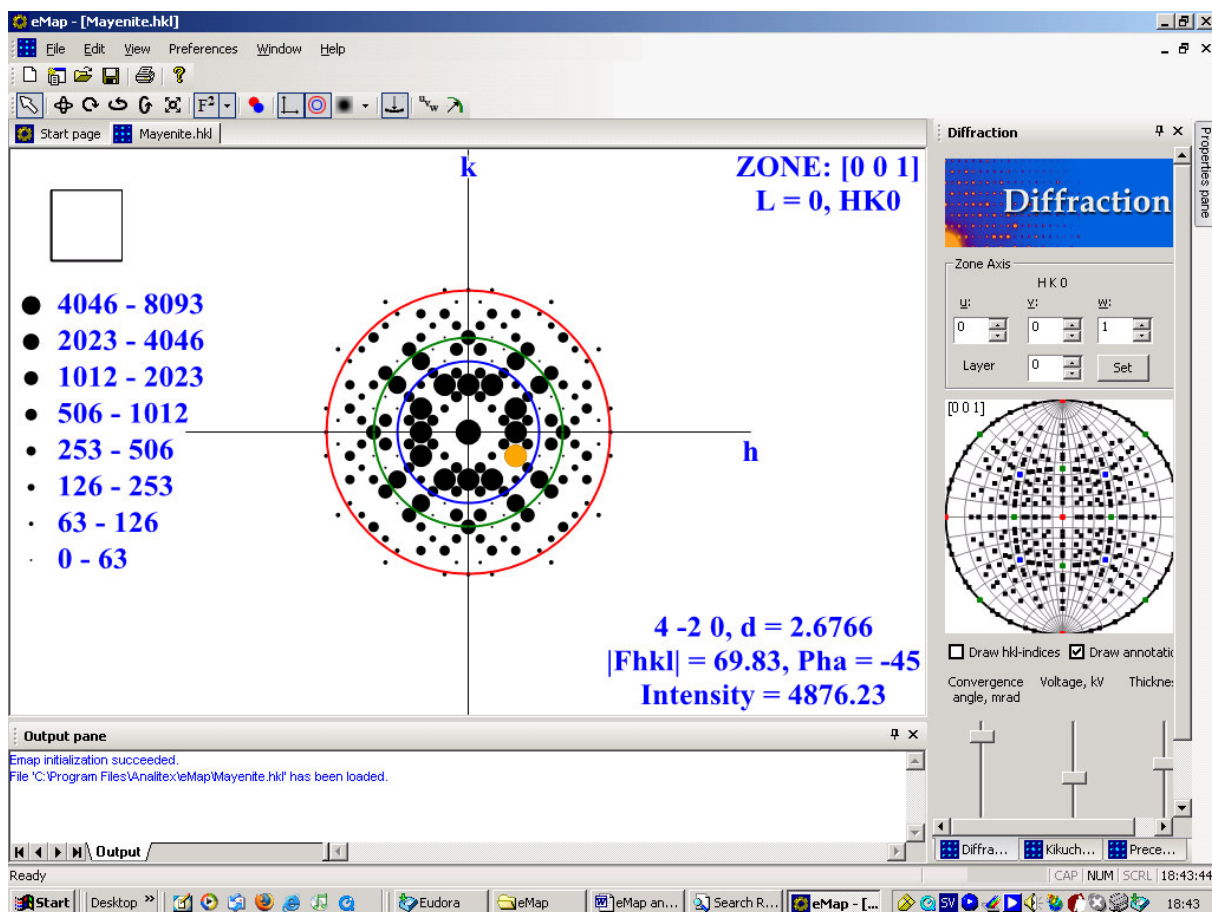
# Simulator: Reciprocal Space Viewer



## 9. Simulator: Reciprocal space viewer

The reciprocal space viewer module is designed for the visualization of reciprocal space. If you have just been running multi-slice simulations, it is best to close down and restart eMap – else the Diffraction panes (seen to the right of the figure below) will not

be displayed. Click on the  icon and then open a file by , for example: **C:/Program files/Analitex/eMap/Examples/mayenite.hkl**



## 9.1. The Reciprocal space viewer toolbar

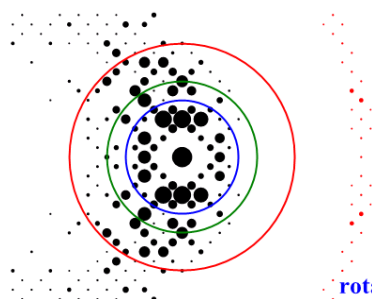
These icons give direct access to many of the functions available under **Preferences**



The normal mouse **pointer for picking reflections** on the diffraction pattern. Placing the mouse pointer over any reflection (high-lighted in yellow) on the main view will display the corresponding reflection information in the right-bottom corner of the view. This information includes the  $hkl$  Miller indices of the reflections, the  $d$ -value in Ångströms, the  $|F_{hkl}|$  amplitude of the reflection, the crystal structure phase and the kinematical Intensity (usually  $|F_{hkl}|^2$ ). Placing the mouse pointer over squared points on the Stereographical projection view (see section 9.3.1 for details) and then pressing **Set** will display the corresponding direction/zone axis;



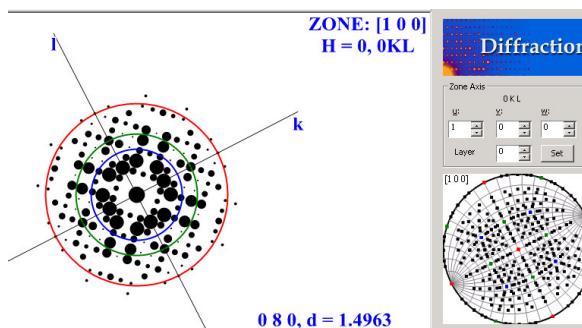
**Free rotation** tool (around x/y axes) for navigation in reciprocal space.



Directly related to the rotation of the crystal in an X-ray diffractometer or an electron microscope. In order to get the correct results, the Ewald sphere toolbar button  should be switched **ON** (see below, page 9.4).



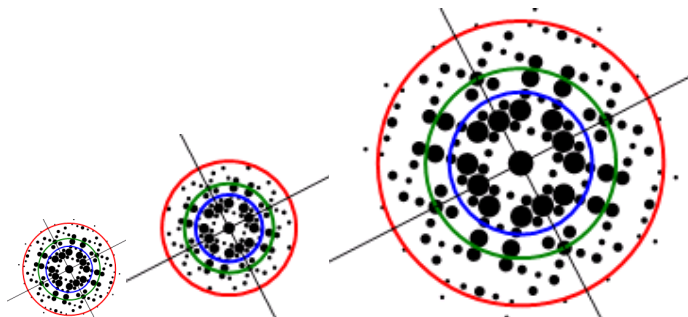
**In-plane rotation** of the diffraction pattern around the  $z$ -axis which always points into the screen. Useful when comparing with an experimental diffraction pattern.



**Rotation about vertical and horizontal axes.**



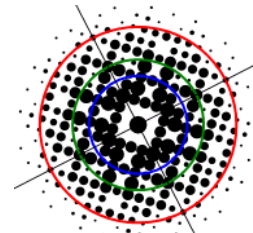
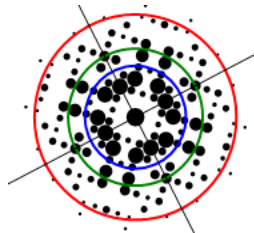
**Mouse zooming**  
Clicking and holding the left mouse button on any point on the main view will fix the starting reference point for the zooming.



The left mouse button should be pressed and held down during the whole zooming procedure. Releasing the left mouse button will stop the zooming. Moving the mouse towards the centre of the diffraction pattern from the starting point will **zoom down** the diffraction pattern, while moving away from the centre will **zoom up**;



**Spot amplitudes mode** – Can be **F** (structure factor amplitudes) or (default) **F<sup>2</sup>** (squared structure factor amplitudes).



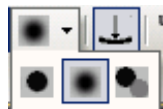
Switches **ON/OFF** the displaying of the in-plane axes.



Switches **ON/OFF** the displaying of the resolution circles.



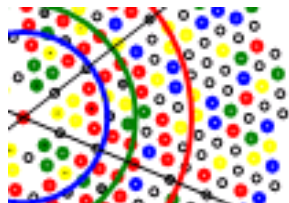
Spot visualization mode. Clicking on the arrow will bring up an extra small toolbar where the user can choose the corresponding mode:



3 different visualization modes are available: 1) solid spot mode 2) Gaussian shape grayscale mode and 3) disk mode.



Switches **ON/OFF** the coloring of reflections. If the button is **ON**, then all reflections will be colored according to their crystallographic phases.

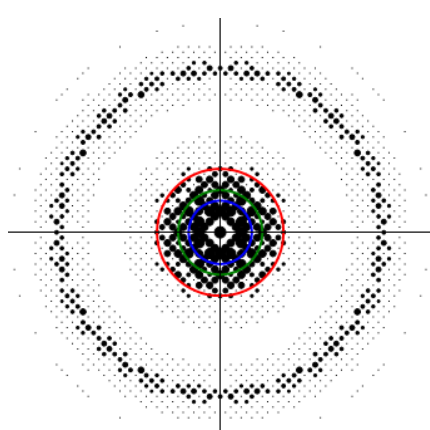
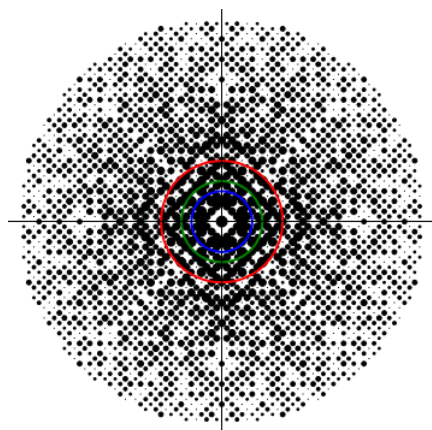


**Red** color corresponds to close to  $0^\circ$ ; **blue** close to  $180^\circ$ , **yellow** close to  $270^\circ$  and **green** close to  $90^\circ$ . Default is **OFF**. Centrosymmetric projections will only show **red** and **blue** reflections.

Note: Coloring is available for solid spot and disk mode only, i.e. not the middle one of the three options: .



Switches **ON/OFF** taking the Ewald sphere into account. If the Ewald sphere is in the **OFF** mode the High Order Laue Zone (HOLZ) reflections cannot be observed, but on the other hand all reflections as far out as they have been calculated are seen for the zero order Laue zone (ZOLZ)



Ewald sphere: OFF: only ZOLZ is seen. Ewald sphere ON: ZOLZ & HOLZ  
Specifies the current zone axis indices



**Zone axis**

u:  v:  w:

Layer  0 K L



Shows the dialog box where the user can specify the radii of the visible resolution circles.

**Resolution circles**

Red:  Å

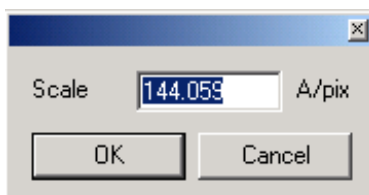
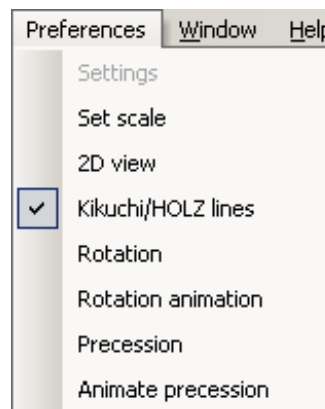
Green:  Å

Blue:  Å

## 9.2. The Preferences menu

In addition to the functions of the Reciprocal Space Viewer toolbar (described above), several other diffraction type visualization modes are available through the **Preferences** of the main menu. These modes are:

- **2D view** Regular 2D diffraction mode (default). Can be controlled using the **Diffraction** pane (see 9.3.1);
- Kikuchi lines. Can be controlled using the **Kikuchi** pane (see 9.3.2);
- Rotation mode and Rotation animation; In rotation mode, the electron beam is rocked back and forth in one direction, or equivalently, the sample is tilted back and forth in that direction. It is fixed to the range  $[-1^\circ$  to  $+1^\circ]$ , with 30 steps.
- Precession mode and Precession animation. Can be controlled using the **Precession** pane (see 9.3.3).

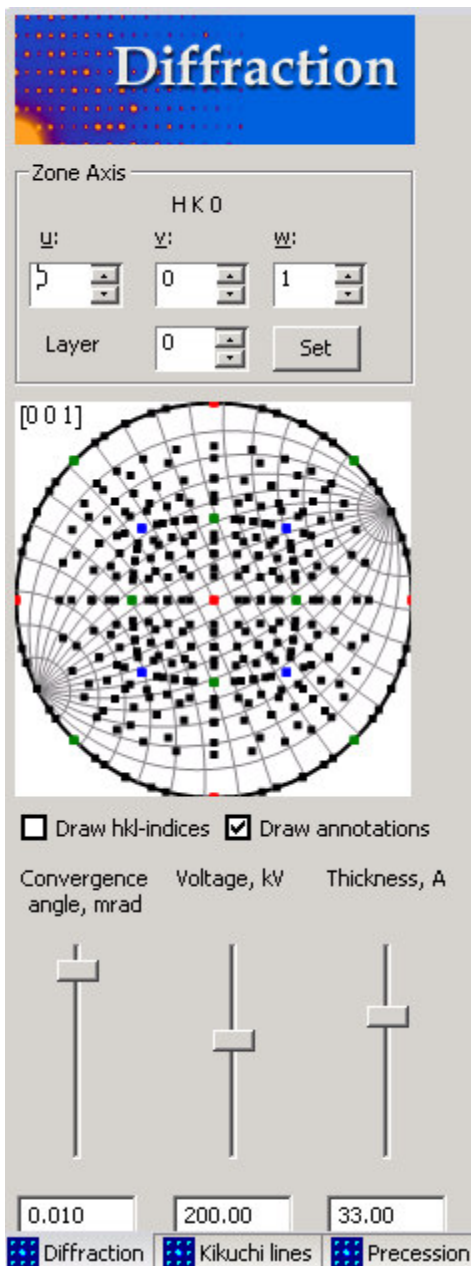


Set scale allows you to scale the simulated diffraction pattern exactly.

### 9.3. Working side pane dialog bars.

The Reciprocal space viewer offers 3 pane dialog bars on the right side (default) of the main view. These bars can be re-attached to any side of the current view or the main window (left or right sides are preferable due to the vertical nature of the dialog bar items placement). Any dialog pane can be closed or hidden any time by using the 2 buttons  in the right top corner of the bar.

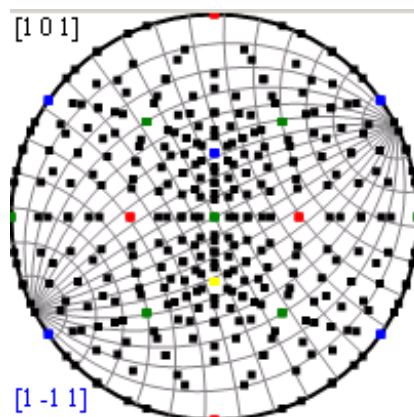
#### 9.3.1. The Diffraction dialog pane



The **Diffraction** dialog pane.

The **Diffraction** dialog pane allows the user to control the current zone axis indices, the Stereographic projection view, show/hide the *hkl* Miller indices for all reflections, show/hide the annotation text, change some parameters of the diffraction pattern such as the beam convergence angle, the voltage (electron diffraction) and the thickness (electron diffraction).

When you move around over the stereographic projection, the index at the bottom left gives the nearest Miller indices. Left-click and the stereographic projection will be reoriented, with that zone axis [given at upper left] at its center.



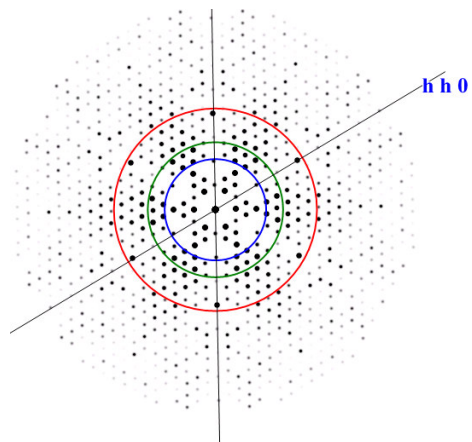
Press  and Simulator will display the electron diffraction pattern along that zone axis:

☒ Draw hkl-indices does just that:

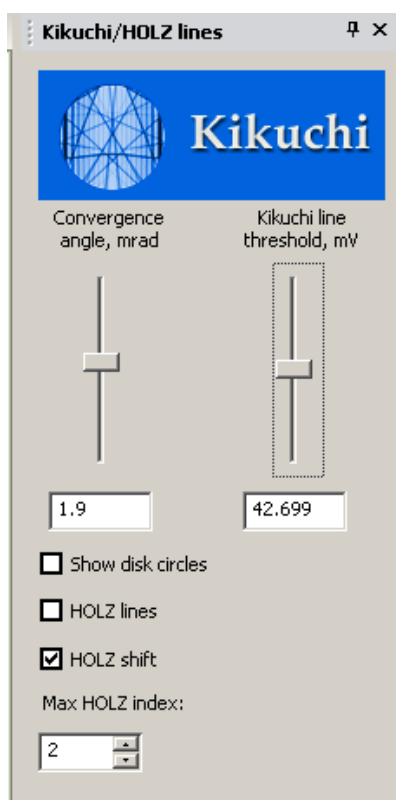


☒ Draw annotations toggles on/off all the descriptive text on the screen:

- 4046 - 8093      -3 -13 0,  $d = 0.8972$
- 2023 - 4046       $|F_{hkl}| = 1.26$ ,  $\text{Pha} = 90$
- 1012 - 2023 ,       $\text{Intensity} = 1.5876$  etc.



### 9.3.2. The Kikuchi dialog pane



Above: **Kikuchi** dialog pane.

Kikuchi lines are useful for crystals with small unit cells, i.e.  $< 8 \text{ \AA}$  or so.



The **Kikuchi** dialog pane allows the user to control the disk size (convergence angle), the threshold for Kikuchi lines and HOLZ lines visualization mode. Checking the **HOLZ lines** will show the central disk (000-spot) enlarged. **NOTE:** in order to observe the HOLZ lines in the 000-disk the following three rules should be satisfied:

1. The Ewald sphere should be switched ON, i.e.



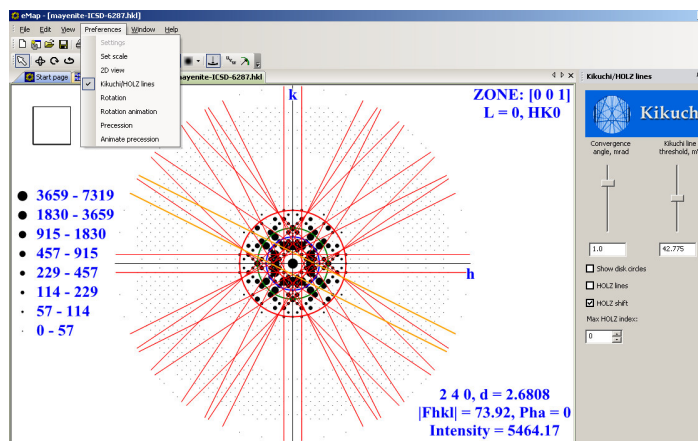
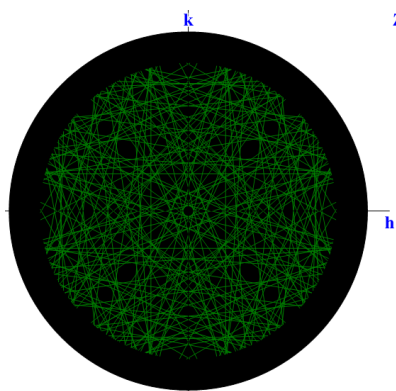
with the frame around it.

2. The calculated diffraction pattern should contain enough spots (enough resolution) so that the Ewald sphere can reach the upper reciprocal layers;

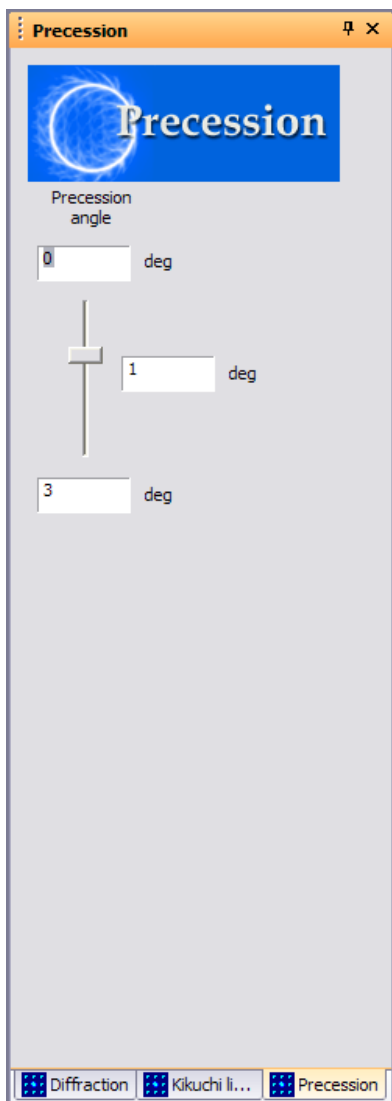
3. The value in the **Max HOLZ index** should be greater than or equal to 0 (default is 0).

Checking/unchecking the **HOLZ shift** will switch ON/OFF the dynamical correction in the calculations of the HOLZ lines positions within the 000-disk which leads to the so-called HOLZ lines shift.

Below: Central beam (highly magnified using )



### 9.3.3. The Precession dialog pane



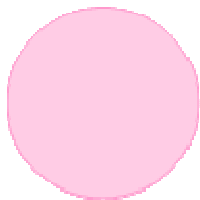
The **Precession** dialog pane allows the user to control the precession angle in **Precession** and **Precession animation** modes. The min and max values of the precession angles can be changed by the user. Here 0 and 3 degrees are used.

☒ **Animate precession** allows you to follow how an electron precession pattern is built up, namely by the successive summation of a large number of different electron diffraction patterns. In order to see the same thing in the electron microscope, the precession must be slowed down to about 1 Hz.

For more information about the precession technique, see the home pages of NanoMEGAS at

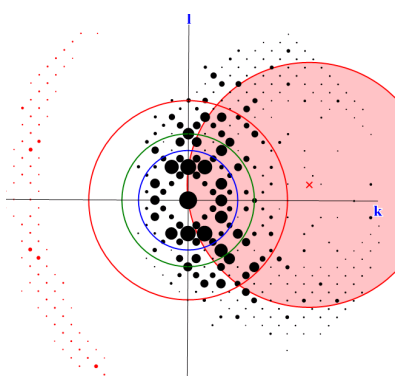
<http://www.nanomegas.com/>

The **Precession** dialog pane.

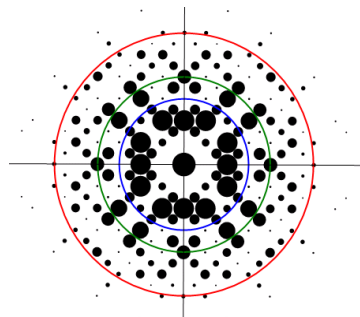


The pink circle is centered on a small red cross, at the distance corresponding to the respective tilt in degrees. It is shown only when the Spot visualization mode is set to

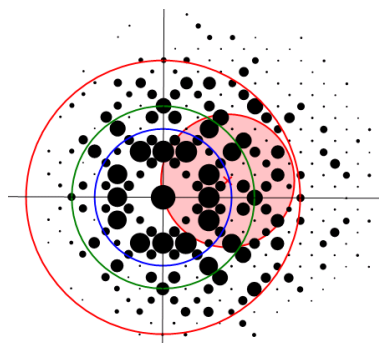
the right: 



**1.** At  $0^\circ$  precession (*right*), the electron diffraction pattern is just the normal selected area electron diffraction (SAED) pattern:



**2.** As the precession angle is increased (*middle*), the momentary electron diffraction pattern looks more and more misaligned. Notice also that the highest resolution reflections are further out with Precession ON.



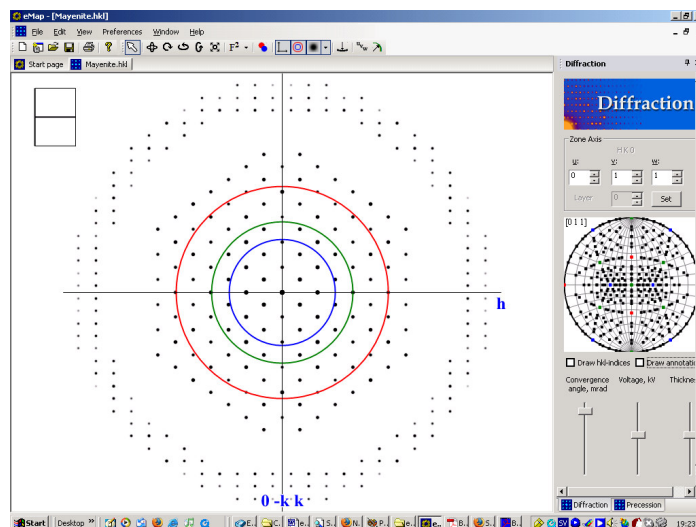
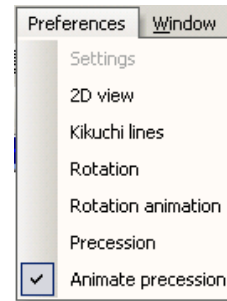
**3.** When the precession angle is even larger (*left*), the FOLZ reflections (marked red here) start to appear at high resolution.

## 9.4. Simulating Precession Patterns

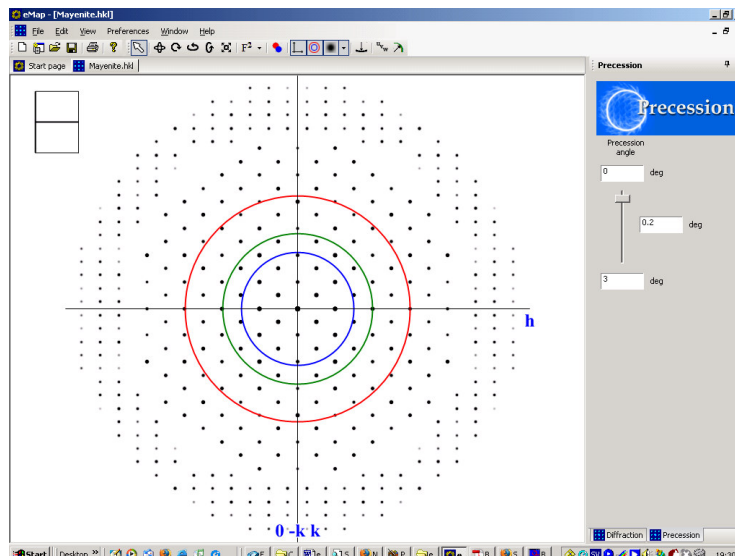
When simulating electron precession patterns, the most clear patterns are obtained when choosing Data type

$Fhkl$ :  $F^2$   $F$  and Rendering Greyscale: 

For the mineral mayenite along the [011] zone axis, the precession pattern at 0° precession is:



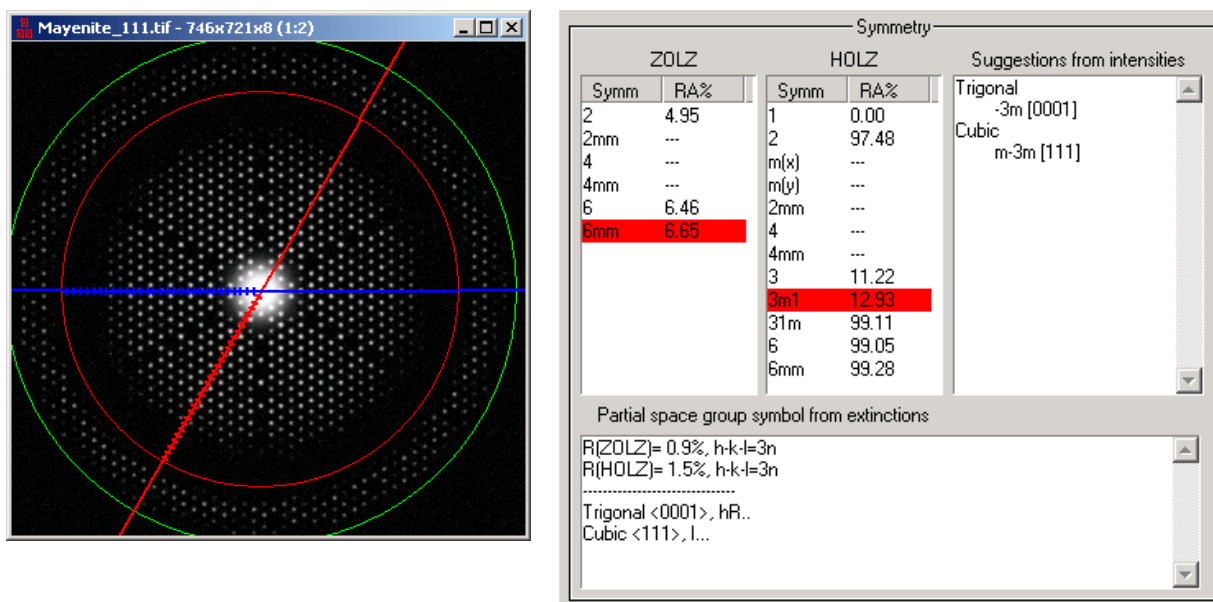
Increasing the precession angle will lead to an expansion of the central ZOLZ and even more marked broadening of the FOLZ circle. Already at 0.2° precession angle the ZOLZ and FOLZ start to merge:



## 9.5. Symmetry determination from precession patterns

The combined information from ZOLZ and FOLZ is very useful for symmetry determination. Notice in the case of mayenite above (space group  $I-43d$ ,  $a = 11.98 \text{ \AA}$ ) that there are twice as many diffraction spots per unit area in the FOLZ ring than in the central ZOLZ are. Notice also that the diffraction spots in the FOLZ are shifted relative those of the ZOLZ. This information can be used to determine the space group, as described in detail by [7]. Experimentally, the symmetry can be determined from such precession patterns by the program **Space Group Determinator** from Calidris, Sollentuna, Sweden. An example is shown below:

## 9.6. Precession electron diffraction pattern of Mayenite along [111]:



Here the symmetry is  $6mm$  in the ZOLZ but only  $3m1$  in the FOLZ. This excludes tetragonal and hexagonal space groups, but allows trigonal and cubic space groups.

The systematic absences (analysed in the bottom window) are only compatible with rhombohedral (in hexagonal setting) [001] and I-centered cubic, along [1 1 1].

## 10. References

1. Z.L. Wang, *Elastic and Inelastic Scattering in Electron Diffraction and Imaging*. Springer. 1995, 476 pp.
2. E.J. Kirkland, *Advanced Computing in Electron Microscopy*. Springer. 1998, 250 pp.
3. **Roger Vincent and Paul Midgley**, Double conical beam-rocking system for measurement of integrated electron diffraction intensities *Ultramicroscopy* **55** (1994) 271-282.
4. **Peter Oleynikov, Sven Hovmöller and Xiaodong Zou**, Precession electron diffraction: observed and calculated intensities. *Ultramicroscopy* **107** (2007), 523-533. A PDF file may be downloaded from the home page of Sven Hovmöller <http://www.fos.su.se/~svenh/index.html>
5. **Peter Oleynikov**, *Exploring reciprocal space – Electron diffraction, texture and precession*, Ph.D. thesis at Stockholm University, Department of Structural Chemistry, 2006. ( 90 pages + 7 papers. Free copies may be obtained from the author or via Calidris.)
6. **Jean Paul Morniroli, A. Redjaïmia and Stavros Nicolopoulos** Contribution of electron precession to the identification of the space group from microdiffraction patterns. *Ultramicroscopy*, **107** (2007) 514-522.
7. **Jean Paul Morniroli and John W. Steeds**, Microdiffraction as a tool for crystal structure identification and determination *Ultramicroscopy* **45** (1992) 219-239.
8. The whole of *Ultramicroscopy* Vol. 107 (2007) issues 6-7, is devoted to the electron precession technique.
9. A large number of references on electron precession can be found at the NanoMEGAS home page <http://www.nanomegas.com/bibliography2.php>